DEVELOPMENT & EVALUATION OF A BENCHTOP WALKAWAY PLATFORM FOR THE AUTOMATED SOLVENT EXTRACTIONS OF CAMBRIDGE FILTER PADS.

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Abstract:

Several documented methodologies require liquid / solid extraction of smoking machine residues from standard size Cambridge filters. The efficiency and reproducibility of such solvent extractions is paramount for optimal results hence the need for operator-independent techniques, and/or appropriate automation.

The requirement for multiple/mixed solvents, variable volumes, choices in vessel types, agitation time and single/multiple sampling and potential 'disposable disc' filtration options must be factored into the project design. Operator preferences to select protocols for wash steps/multiple washes are also essential. Researchers seeking to analyze cryogenically ground tobacco leaf samples have similarly stringent demands.

Our poster describes the development & usage of an automated Extraction Station based around standard and customary approaches, but utilizing a robotic platform to ensure Uniform Sample History, and an ongoing chain-of-operations database for all samples thus prepared.

Overview:

The ISO 10315 : 2000 / ISO 10362-1:1999 and CORESTA methods for Nicotine & Water Content of smoke condensates via GC are well known industry wide.

To recap, cigarettes are smoked by machine and the condensates collected on a 'Cambridge' Filter. This filter is used to wipe dry the inside of the holder device, and then carefully inserted into a conical flask to which anhydrous solvent is added and the flask is stoppered. Flasks are shaken (usually on orbital agitation device) for a preprogrammed period at a medium speed that doesn't degrade the integrity of the filters, at a specified stroke length. Following this, Aliquots are taken and dispensed into GC vials which should be capped immediately.

The GC assays for Water or Nicotine or both analytes are documented in the referenced methods.

Our goal was to develop an automated system to allow a technician 'walkaway time' - thus reducing need for headcount, and providing an auditable sample history, and further ensuring that each sample is treated uniformly to help ensure better precision of results.

Specific issues to be addressed:

1. Compliance – the System must be intrinsically compliant with published methods.
2. Capacity – To ensure there is at least space for 15 flasks on the shaker deck (2 batches incorporate both samples plus controls from 2 batches from Smoking Machine). Ensure the robot can access each of the flasks and pick/hold the stoppers whilst dispensing and/or sampling. In contrast the shaker must be sufficiently compact to fit on robotic deck.
3. Shaker Homing – The robot needs to know where the flask openings are exactly, and the answers are to either perfect a foolproof homing device for the shaker OR invest in expensive robot vision.
4. Speed of fluidics – When one is aiming at uniform sample history and is competing with manual operations, the time taken for a large volume (>10mL capacity) syringe pump to fill and dispense becomes significant. A better answer was required and it had to meet or exceed 'repeat dispenser' accuracies and performance.
5. If we are to use a common cannula between samples there must be a user programmable method for repeat washes of inner/outer whetted surfaces, using solvent (mix) of choice.
6. Flask seals – Whether using ground glass or polymer stoppers, robots generally do not have an intrinsic ability to 'wiggle/twist' a stopper during removal. The stoppers must be vapor tight, and yet robot compatible. (A crimp cap/septum solution was deemed unacceptable)
7. Filtration option – sometimes filter pads fall apart and filtration of the extraction fluid has to be an option.
8. Capping of GC vials – should also be an available option, for obvious reasons.
9. The robotic system should offer a means of alerting the Operator that the run is completed.
10. All variables must be user–selectable, and yet the user interface should be both GLP and 21CFR11 compliance capable.

Discussion, Recommendations & Conclusions:

Obviously, we are pleased to report that our development project concludes positively. Once installed, the machines have worked flawlessly electromechanically, and have provided their Operators with a more productive laboratory workplace.

Our intent to show improvements in Standard Deviation numbers between Automated vs Manual remains, but the early data really only points to equivalency. Over time, we plan to revisit this question.

There are some remaining concerns of over/under-reporting depending upon which cannula type is employed (for the two robotic systems in this study, they are slightly different…the better results are from the unit fitted with a Teflon coated Cannula). The authors intend to engage in further work to confirm this finding, and retrofit extant installations as needed.

Further – we seek additional partnering to engage in logical extensions of this work to investigate other analytes and possibly biological materials.


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[System Collected Data]
================================================================
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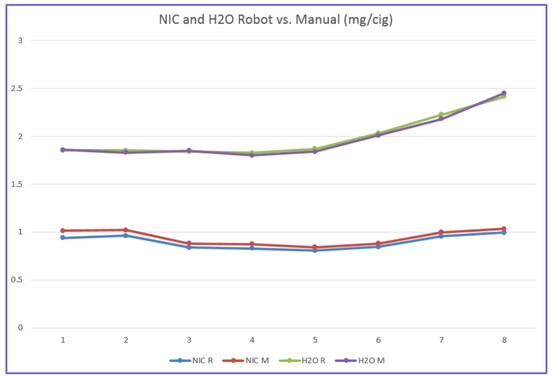
Sample Pos  Extraction Volume (mL)  Transfer Volume (mL)
001         50.0000                 1.5000
002         50.0000                 1.5000
003         50.0000                 1.5000
004         50.0000                 1.5000
005         50.0000                 1.5000
006         50.0000                 1.5000
007         50.0000                 1.5000
008         50.0000                 1.5000
009         50.0000                 1.5000
010         50.0000                 1.5000
011         50.0000                 1.5000
012         50.0000                 1.5000
013         50.0000                 1.5000
014         50.0000                 1.5000
015         50.0000                 1.5000
```

Robot automatically emails Operator when each process reports.

Data



N.B. H2O data and NIC data from different sample cadres.

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