New research could improve pharmaceuticals testing

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A UT Arlington chemistry professor, renowned for his work in the area of chemical separations, is leading an effort to find a more accurate way to measure water content in pharmaceuticals – a major quality issue for drug manufacturers.

Daniel W. Armstrong, UT Arlington's Robert A. Welch Chair in Chemistry, says the new technique could be 100 times more sensitive than one of the most popular current methods.

"The analysis for water in many consumer products, including drugs, is one of the most required tests done in the world," said Armstrong. "Current methods have many shortcomings, including poor sensitivity and reproducibility; they cannot be used for all products and they can be time consuming. I believe our new 'ionic liquid' method offers improvements in all these areas."

Armstrong and two graduate students recently wrote about their new research in a paper that will be in the June issue of the *Journal of Pharmaceutical and Biomedical Analysis*. The publication describes using headspace gas chromatography and an ionic liquid gas chromatography column Armstrong's lab developed to measure moisture content in active pharmaceutical ingredients such as ibuprofen, tetracycline and ephedrine. Water content can affect the stability and shelf life of a drug and, when it is too high, cause microbial growth, according to the paper.

Chromatography is the process of separating a chemical mixture to

measure its components. Headspace gas chromatography, or HSGC, involves the measuring of volatile analytes, or chemical components, as they diffuse into a "headspace" at the top of a tube of sample, including solids. The new method combines HSGC with the use of ionic liquids. Ionic liquids consist of a mixture of positively and negatively charged molecules. They have a variety of advantages as solvents.

Currently, two methods are most commonly used to measure moisture content. One is called weight loss on drying, or LOD; the other is called Karl Fischer Titration, or KFT. The newly described work from Armstrong's lab is useful on more types of drug ingredients than LOD and is more than 100 times more sensitive in some cases than KFT. It also can be used for much smaller samples and be automated, the paper said.

Graduate student Lillian A. Frink is lead author on the paper and Choyce A. Weatherly, also a graduate student in Armstong's lab, is a co-author.

"We think industry will utilize this method based on its broad applicability, its high accuracy, and the sample size requirements," said Frink. "It also doesn't have side-reactions like current methods, which make them inaccurate."

Armstrong has been a leader in the characterizing and synthesizing ionic liquids. He also holds patents on several open tubular capillary gas chromatography columns that utilize ionic liquids, including those used in the current research.

More information: The newly published paper is called "Water determination in active pharmaceutical ingredients using ionic liquid headspace gas chromatography and two different detection protocols." It is available here: http://www.ncbi.nlm.nih.gov/pubmed/24561336.

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