B164 The Use of Wick Evaporation With Wicking Bottles to Prepare Crystals for Micro FT/IR Spectroscopy

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After attending this presentation, attendees will learn an improved method for preparing small crystals of purified materials suitable for Infrared Microspectroscopy.

This presentation will impact the forensic community and/or humanity by demonstrating a simple way to isolate or purify many different types of trace materials for unambiguous identification by infrared spec- troscopy. It is particularly appropriate for moisture sensitive compounds. Examples will be given of drugs and explosives for which this method should be advantageous.

The use of a little know technique[1] for recovering small amounts of soluble material from a much larger volume of insoluble material called wick evaporation[2] has been reported That paper used the method to recover purified LSD and iso-LSD, in the form of tiny crystals, from preparative thin layer chromatography. These crystals were shown to provide excellent Infrared spectra using a micro FT/IR spectrometer. A modification of this technique which makes it simpler to use and much more versatile is reported here.

By carrying out the wick evaporation in a closed system, which is referred to as a wicking bottle, a much wider range of solvents and conve- niently work with moisture or air sensitive materials can be used. The wicking is done as before in a shell vial. General procedure is to have from 500 to 1000 micrograms of target compound and to add about one milliliter of wicking solvent. The vial is placed in a small bottle (a four ounce glass bottle is convenient) containing five to ten grams of finely divided silica which has been activated by heating to about 150 degrees for a number of hours (usually about twenty-four) to drive off adsorbed solvent and activate the silica. Thus the wicking is done in a closed system and the crystals are produced on the portion of the wick sticking out of the vial, usually toward the end. The crystals are kept in a dry atmosphere until removed or the vial and wick can be removed and placed in a dessicator. The wicking bottle is regenerated to be used again by placing it in a 150 degree oven overnight or until it is needed again.

This technique allows one to use wicking solvents covering a wide range of volatility and polarity. Solvents such as non-polar hydrocarbons like hexane to more polar materials such ethyl acetate, tetrahydrofuran and even methyl or ethyl alcohol have been successfully used. The volatility range was severely limited when the wicking was done in the open atmos- phere in a hood, because in humid weather evaporative cooling condensed ice crystals on the end of the wick and interfered with the desired crystal- lization.

One can wick organic bases that are not normally easily crystallized solids by converting them to their hydrochloride salt before wicking. Many of these salts wick well with THF, ethanol or methanol. Further very hydroscopic materials such as ammonium nitrate and sodium gammahy- droxybuterate, which can absorb enough water from the atmosphere to dis- solve themselves in humid weather, can be wicked to produce crystalline materials for infrared spectroscopy.

The simplicity of the method was increased by using twine for wicks. In the first paper the wicks were made by twisting a small piece of glass wool into a wick. Although this is not difficult, snipping off a piece of braided twine is quicker, easier and one has more uniform wicks. One can purchase nylon or polypropylene twine at most hardware stores and of course cotton is also available. Nylon was used for most of the wicks but the polypropylene seemed to work as well. Four examples will demonstrate the versatility of this method: Separation of cocaine base and cocaine hydrochloride, recovery of sodium gammahydroxybuterate from solution containing gammabuterolactone and salt, recovery of materials from preparative thin layer chromatography and recovery of ammonium nitrate from a simulated debris mixture.

References:

1. Chriswell, C.D. and Markusqewski, R., "Wick Evaporation: A Technique for the Isolation of Soluble Analytes from volatile sol- vents", Analytical Chemistry, Vol. 60, 1988, pp 508-509.
2. Harris, H.A. and Kane, T., "A Method for Identification of Lysergic Acid Diethylamide (LSD) Using a Microscope Sampling Device with Fourier Transform Infrared (FT/IR) Spectroscopy," Journal of Forensic Sciences, JFSCA, Vol 35, No. 4, July 1991, pp1186-1191.

Sample Preparation, Infrared Spectroscopy, Purification

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