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AN AUTOMATED METHOD TO CLEANUP AND DETECT PULPWASH ADULTERATION OF ORANGE JUICE CONCENTRATES.

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Sodium benzoate is added to orange pulpwash as a tracer to help determine if orange pulpwash has been used to adulterate frozen orange juice concentrate. The procedure used, developed by J. Fisher (1) of the Florida Department of Citrus consists of reconstitution, centrifugation, filtration of supernatant, solid phase extraction cleanup, and analysis by HPLC.

An automated method has been developed to perform the solid phase extraction cleanup and HPLC analysis witout operator intervention. This method employs high and low pressure valve switching to perform the automated analysis.

After initial sample preparation (centrifugation), the orange juice supernatant is placed into vials and loaded into a WISP(TM) auto-injector. The WISP(TM) injects 10ul into a flowing stream of 1% phosphoric acid which deposits the sample onto the head of a C18 Guard-Pak(TM) pre column. The pre-column is connected to one of the six port high pressure switching valves of the Waters Automated Valve Station(TM). This valve first directs a 4 ml wash of 1% phosphoric acid across the pre column and off to waste. Next a 2 ml was of a NaOH solution (0.25 g/liter) is passed over the pre-column and also directed to waste. Finally, a 1 ml "plug" of acetonitrile/NaOH solution is introduced into the pre-column and the high pressure valve is reversed to backflush the analyte onto the head of the analytical column. The pH changes listed above are necessary to insure that the sodium benzoate is converted to the acid form and is retained by the pre-column until elution is desired. The analytical mobile phase is adjusted to a pH of 2.5 to compensate the relatively high pH of the sample.

The equipment utilized to perform this automated process consists of the Waters Automated Valve Station(TM), the M501 and M590 Solvent Delivery Systems, an M680 Automated Gradient Controller, a WISP(TM) auto-injector, and RCM-100, a Guard-Pak(TM) module, a C18 Guard-Pak(TM), a C18 Radial-Pak(TM) cartridge, and M740 Data Module and an M481 variable UV detector. (All from Waters Associates, Milford, MA)

The recoveries from this automated procedure are equal to or better than those delivered by the manual clean-up and analysis. Consistency of results is better for the automated analysis. Additional benefits of the automated procedure are an approximate 50% time reduction per sample and the ability to clean-up and analyze 96 samples unattended.

(1) Fisher, J., J. Agric. Food Chem. 31, 66-68, (1983)