〔J. Chromatogr., **464**, 172 (1989)〕

Application of deactivated metal capillaries to the analysis of solvents in varnish.

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Tracing of the decrease in solvents in varnish was carried out satisfactorily by using a deactivated metal capillary tube as a cold trap and a deactivated metal capillary column as a GC column. The metallic cold trap was rapidly heated directly with an electric current and the capillary column was removed from the oven while it was connected to the gas chromatograph to be chilled to obtain the peaks of the separated low volatile solvents and after an appropriate time it was replaced in the oven for further temperature programming. The deactivated metal capillary column withstood continuous use at 450°C in the case of polymethylsiloxane as the stationary phase and retained sufficient mechanical strength for column exchange after use. Thus the column is advantageous as a high-temperature GC column compared with a fused-silica capillary.

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Improved method for the separation of methylolmelamines by high-performance liquid chromatography.

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The present paper describes a rapid and sensitive reversed-phase HPLC assay which permits the microanalysis of nine methylolmelamines. HPLC conditions: column; ODS-5 (250 × 4.6 mm I. D.), mobile phases;acetonitrile-acetic acid-water (5: 0.5: 94.5, v/v/v) for the separation of highly substituted methylolmelamines and (1:0.5:98.5, v/v/v) mixture for low substituted methylolmelamines, 1.8 ml/min, room temperature, detector; UV (235 nm). The present method was applied to the determination of melamine and methylolmelamines in extracts from cups made of melamine resin, and exudation melamine and monomethylolmelamine was observed. The presence of monomethylolmelamine was identified for the first time in extracts from cups made of melamine resin.

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High-performance liquid chromatographic determination of malonaldehyde in serum.

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The present paper describes a HPLC method for assaying low levels of malonaldehyde in serum. To 0.4 ml of serum in a 1.5-ml centrifuge tube were added 0.1 ml of 1 % sodium hydroxide solution and 0.5 ml of acetonitrile, and the well mixed solution was incubated at 60°C for 30 min in a heating bath. After centrifugation at 2000g, 0.1 ml of 1 M hydrochloric acid was added to 0.5 ml of the supernatant and the mixture was reacted with 0.2 ml of the 2,4-dinitrophenyl hydrazine solution containing an appropriate amount of 2-nitroresorcinol as internal standard at room temperature for 1 h. After centrifugation, if necessary, an aliquot of the reaction mixture was injected into the HPLC column. HPLC conditions:column; ODS-5 (250 × 4.6 mm I.D.), mobile phase; CH_3CN-0.01 M HCl (45:55,v/v), 1.5 ml/min, room temperature, detector; UV(310 nm).