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Gas–liquid chromatographic analysis of small amounts of formamide

Small amounts of formamide (above 1000 p.p.m.) affect the catalytic hydrogenation of adiponitrile. On a polar stationary phase such as Carbowax 20 M or polyethylene glycol adipate, good separation of these two compounds can be easily obtained, but if the stationary phase is poured on the common diatomite aggregates, even when they had been treated with TMS or DMCS, the lowest detectable amount is too high because of the irreversible adsorption of the solute on the active sites of the support.

Because of the interference due to various cyanoethylation byproducts, it is impossible to use another analytical method such as non-aqueous solvent titration or IR spectrophotometry.

In gas chromatography, the flame ionisation detector is less sensitive than the thermal conductivity detector and the electron capture detector is rapidly overloaded by the high content of adiponitrile in the injected samples.

To avoid irreversible adsorption of the support, we have used a specially inert material, described previously by SAINT YRIEIX AND LESIMPLE[1], *viz.* thermally treated and screened Teflon®.

Experimental

To prepare the solid support, 10 parts (by weight) of Chromosorb T, 40–60 mesh, are mixed carefully with 85 parts of dried sodium chloride (C.P. grade), and the mixture is placed in an oven at 310–320° for 6 h. This temperature range is carefully controlled: below 310°, no difference can be observed in the behaviour of the treated Teflon particles, and above 320°, the specific area of the Teflon and consequently the amount of stationary phase that can be poured on it decrease rapidly.

After the thermal treatment, the mixture is cooled, washed first with water to eliminate the sodium chloride and then with acetone, and finally dried in an oven at 110°.

Twenty parts (by weight) of Carbowax 20 M are dissolved in chloroform; eighty parts of the treated Chromosorb T are poured in and the solvent is allowed to evaporate slowly with gentle agitation. After drying in an oven at 100°, the material is finally screened between the 40 and 60 mesh sieves. The filling of the column is very easy as the electrostatic effects have completely disappeared so that the Teflon beads never aggregate.

The following experimental conditions are used: apparatus: FM 720; column: stainless steel tube, length 0.5 m, I.D. 1/4 in., Carbowax 20 M, 20% on treated Chromosorb T, 40–60 mesh; temperatures: column 200° isothermal; injector 260°; detector 240°; carrier gas: helium (flow rate 40 ml/min); sample: 25 μl; detector: thermal conductivity, tungsten filaments; current 200 mA; recorder: Honeywell Brown, 1 mV.

Results

Retention volumes corrected for dead volume were measured and compared with that of *m*-cresol (used as external standard): formamide (0.45), *m*-cresol (1.00), adiponitrile (1.95).

For quantitative analysis, the external standard addition method was chosen with *m*-cresol as reference compound. Standardisation was carried out with artificial solutions containing 40 p.p.m. to 1% of formamide. Statistical analysis gives a reproducibility of 5% (relative value). The detection limit is about 20 p.p.m.

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1 A. SAINT YRIEIX AND C. LESIMPLE, *Bull. Soc. Chim. Fr.*, II (1967) 4365.

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