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Note

Nickel(II)-exchanged molecular sieve 13X as a column packing for the gas chromatographic determination of thiophene in benzene with flame ionization detection

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The determination of small amounts of thiophene in benzene is difficult owing to their similar physico-chemical properties. Several gas chromatographic (GC) methods have been developed employing either selective detectors[1-3] or other appropriate systems[4,5]. Sulphur-specific detectors are still very expensive and only a few gas chromatographs are now equipped with these devices. When a non-selective detector is used, the long tail of the benzene signal overlaps the small thiophene peak. In order to overcome this problem, some workers used a combination of several columns and valves for venting the major portion of benzene to the atmosphere prior to the analysis. However, the results were not satisfactory, mainly owing to the necessity to use very complicated systems.

In this work the possibility of using GC with flame ionization detection for the determination of thiophene in benzene with a nickel(II)-exchanged molecular sieve as the column packing material was studied.

EXPERIMENTAL

Materials

Solutions of thiophene in benzene (1–100 ppm) were prepared by weighing. Sulphur-free benzene was obtained by boiling analytical-reagent grade benzene (POCh, Gliwice, Poland) with freshly prepared Raney nickel. Analytical-reagent grade thiophene (POCh) was additionally purified by distillation.

The nickel(II)-exchanged molecular sieve 13X employed as a column packing was prepared as described earlier[6]. This material was chosen after our earlier evaluation of some modified molecular sieves 13X for the separation of thiophene and benzene[6]. Of all the molecular sieves investigated the most useful relative retentions of these compounds were observed on the nickel-exchanged sieve.

Chromatographic conditions

All analyses were carried out on a Hewlett-Packard Model 5830A gas chromatograph equipped with a flame ionization detector. The molecular sieve (400 mg) was packed into a 20 cm × 3 mm O.D. stainless-steel column. The conditions of the analysis are given in the caption of Fig. 1.
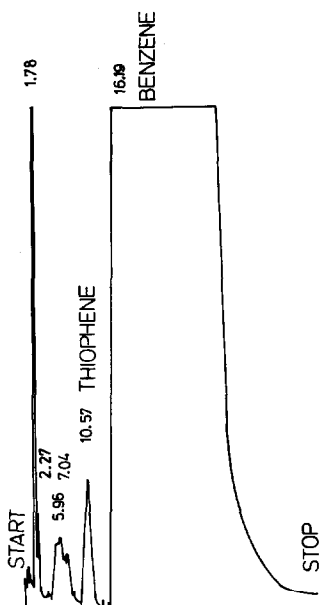
Fig. 1. Chromatogram of thiophene (retention time 10.57 min) in benzene (retention time 16.19 min). Sample: 1 μl of a 15 ppm solution. Column: 20 cm × 3 mm O.D., stainless steel, packed with 400 mg of nickel(II)-exchanged molecular sieve 13X. Column temperature, 210°C; injector temperature, 250°C; detector temperature, 200°C; carrier gas, argon at 36 cm³/min; attenuation, 16; chart speed, 0.2 cm/min.

RESULTS AND DISCUSSION

A typical chromatogram of thiophene in benzene obtained using the nickel-exchanged molecular sieve as column packing is presented in Fig. 1. It can be seen that this molecular sieve, even in small amounts, separates thiophene and benzene completely, thus permitting the utilization of any gas chromatograph equipped with a flame ionization detector. Therefore, the main advantage of this procedure is the simplicity of the apparatus required.

It should be noted that, compared with typical stationary phases, the order of elution of thiophene and benzene is reversed. Benzene is retained more strongly than thiophene, which is especially important in trace analyses.

The procedure has been employed on a routine basis in the range 10–100 ppm (w/w) of thiophene. It is possible to use it in the range 1–10 ppm, but then it is necessary to decrease the attenuation for the thiophene peak and to work close to the detection limit of the detector, which can sometimes be troublesome.

The method should be especially useful for the analysis of technical-grade benzene. The column operated for one month without a noticeable decrease in the quality of separation. The main disadvantage is the long analysis time, which depends on the concentration of thiophene in benzene.

ACKNOWLEDGEMENT

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