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Note

Simple, direct gas chromatography–mass spectrometry interface for the ion trap detector

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Since its introduction in 1983[1,2], the utility of the Finnigan-MAT ion trap detector for gas chromatography–mass spectrometry (GC–MS) has been augmented by the addition of automatic gain control[3] and chemical ionization capability[4]. However, the GC–MS interface has remained unchanged and consists of an approximately 4-ft. long flexible transfer line designed for use as an open-split interface[5]. In an open-split interface, the exit of the capillary GC column is designed to operate at atmospheric pressure. The deactivated fused-silica restrictor housed within the transfer line connects the capillary GC column to the mass spectrometer and provides the pressure drop required by the mass spectrometer's vacuum system. For our work in pesticide residue analysis, we often employ short (3–4 m) capillary GC columns for rapid analysis of labile pesticides and their metabolites[6]. Such analyses can best be accomplished with the exit end of the capillary column directly coupled to the MS vacuum in conjunction with high carrier gas linear velocities[7–9]. For use as a direct GC–MS interface, the existing interface is cumbersome to work with, particularly when changing columns, and results in a significant portion of a short capillary column residing within the transfer line.

In this paper, we report on a simple, direct GC–MS interface of less than 1 ft. in length which provides a line-of-sight path for the capillary column enabling direct insertion of the column outlet into the ion trap. The interface was designed to facilitate insertion and removal of the column from the ion trap and can be readily constructed and retrofitted to existing ion trap detectors. The new interface has made possible the GC–MS confirmatory analysis of terbufos, terbufos sulfoxide and terbufos sulfone at residue (1 ng) levels, an analysis previously performed[6] on a Finnigan-MAT TSQ-70.

EXPERIMENTAL

Apparatus

A drawing of the interface is shown in Fig. 1. The transfer line consists of an empty stainless-steel liquid chromatography column (25 cm × 2.1 mm I.D. × 1/4 in. O.D.; Part No. 5-9127, Supelco, Bellefonte, PA, U.S.A.) and is connected to the ion

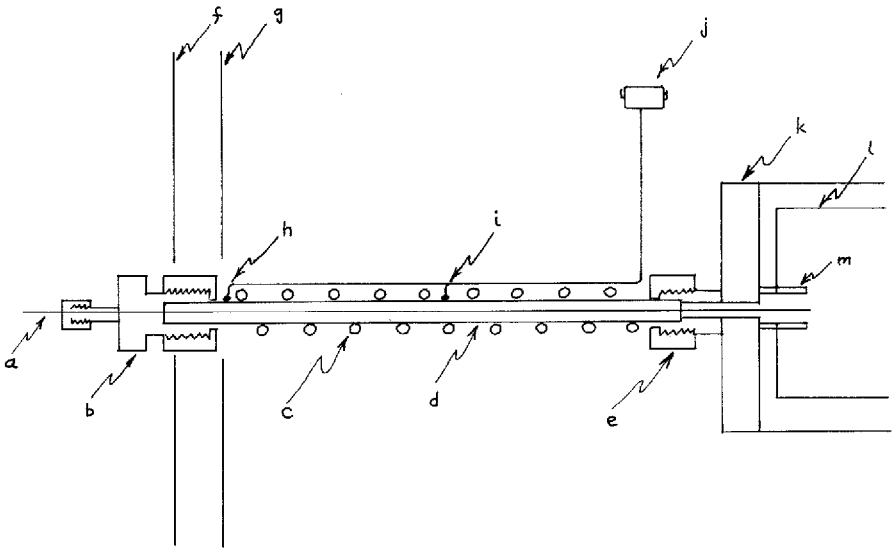

Fig. 1. Drawing of the direct GC–MS interface. a = Capillary GC column; b = 1/4 in.-to-1/16 in. Swagelok reducing union; c = coiled cable heater; d = 1/4 in. O.D. stainless-steel column; e = 9/16 in. Swagelok nut; f = GC oven inner wall; g = GC oven outer wall; h = "open-split" thermocouple; i = "transfer line" thermocouple; j = Burndy connector; k = ion trap manifold; l = ion trap; m = Rulon spacer.

trap manifold by a 9/16-in. Swagelok nut and graphitized vespel ferrule. A 1/4 in.-to-1/16 in. Swagelok reducing union connects the transfer line to the capillary GC column and provides the vacuum seal for the mass spectrometer. Removing the 5/16-in. Swagelok nut on the reducing union is all that is required for changing capillary GC columns interfaced to the mass spectrometer. The overall length of the transfer line from the back of 5/16-in. Swagelok nut to the end of the 1/4 in. O.D. tubing is 270 mm. The distance from the back of the 5/16-in. nut to the inside wall of the ion trap manifold is 283 mm. Combining the latter value with a measured length of 15 mm for the Rulon guide within the ion trap itself yields an overall length of 298 mm of capillary GC column to reach from the back of the 5/16-in. nut to the outlet of the Rulon guide within the ion trap.

Heating of the transfer line is provided by a coiled cable heater (24 in., 120 V, 225 W, 0.25 in. coil I.D., 9.00 in. coil width, standard 2A lead orientation; Part No. 62H24A6X, Watlow Electric, St. Louis, MO, U.S.A.) and uses the same heater power supply as used by the manufacturer's "transfer line" heater. The "transfer line" thermocouple (iron–constantan, Part No. TC-GG-J-20-36-STD, Omega Engineering, Stamford, CT, U.S.A.) is attached with high-temperature cement (Omega Bond 200, Omega Engineering) approximately midway along the transfer line and equally spaced between two turns of the coiled cable heater. The "open-split" thermocouple is attached in the same manner next to the reducing union. The heater and thermocouple leads are attached to a Burndy connector used by the manufacturer for the ion trap detector "transfer line" (Part No. 00004-22050, Finnigan-MAT, San Jose, CA, U.S.A.). The transfer line is insulated with flexible ceramic tape (Part No. 395-41, Cotronics Corp., Brooklyn, NY, U.S.A.) and two layers (1/2 in. I.D. × 1/8 in. wall and

1 in. I.D. × 1/8 in. wall) of braided fiberglass sleeving (Flextex, Ambler, PA, U.S.A.). The "exit nozzle" heater is placed on top of the 9/16-in. Swagelok nut at the ion trap manifold and the "exit nozzle" thermocouple is positioned between the nut and the heater.

The gas chromatograph is a Varian 3500 and is sited with the right side (viewed from the front) facing the rear of the ion trap detector. A 1 3/4 in. diameter hole through the right side panel and a 3/4 in. diameter hole through the GC oven wall are drilled on-line with the GC inlet port on the ion trap manifold. Following attachment of the transfer line to the ion trap manifold, the holes in the GC are slid over the interface until the 9/16-in. nut on the reducing union rests on the inside wall of the column oven. This positions the side of the GC approximately 5 in. from the rear of the ion trap detector and allows adequate room for air intake and exhaust to cool the electronics of the mass spectrometer.

Materials

Terbufos, terbufos sulfoxide and terbufos sulfone were obtained from American Cyanamid Company (Princeton, NJ, U.S.A.). Lauryl laurate was purchased from Pfaltz & Bauer (Stamford, CT, U.S.A.).

RESULTS AND DISCUSSION

The first test of the direct GC–MS interface for a difficult pesticide residue analysis was to determine if chromatography obtained on terbufos and its oxidative metabolites previously performed on a TSQ-70 could be replicated with the ion trap detector. Terbufos sulfoxide is an organophosphorous pesticide metabolite which is notoriously difficult to chromatograph[10,11]. A short capillary GC column from injector to detector, high carrier gas linear velocities, and rapid temperature programming of the column oven (30°C/min) are required for a successful analysis[6]. Using the same chromatographic conditions as employed previously, the chromatography of terbufos and its oxidative metabolites excellently matches that reported in the literature. As shown in Fig. 2, excellent peak symmetry is evident for each analyte

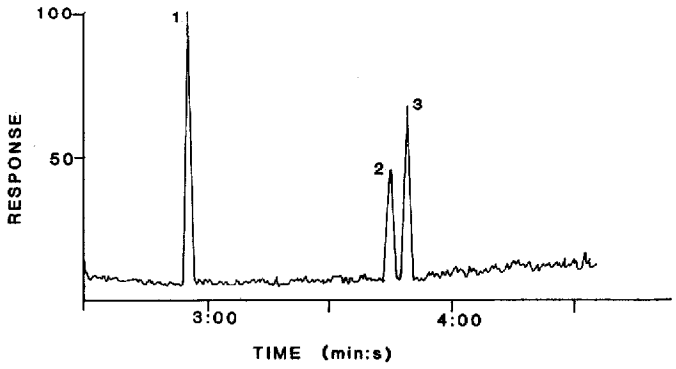

Fig. 2. Total ion current chromatogram from 1 ng each of terbufos (1), terbufos sulfoxide (2), and terbufos sulfone (3) using the direct GC–MS interface.

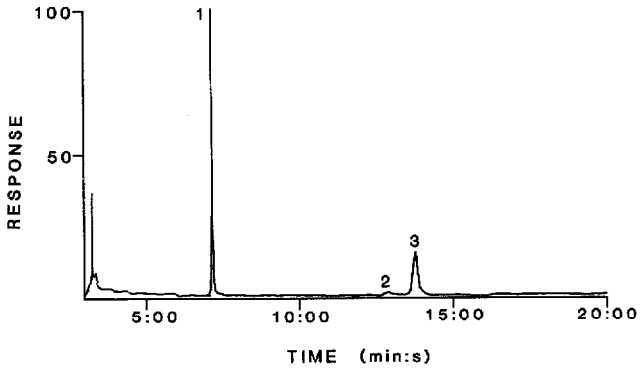

Fig. 3. Total ion current chromatogram from 100 ng each of terbufos (1), terbufos sulfoxide (2), and terbufos sulfone (3) using the manufacturer's open-split interface.

at the 1-ng level. The electron impact mass spectra of the analytes compare favorably with those in the literature[10,11].

Earlier attempts to replicate this analysis on the ion trap detector with the manufacturer's open-split interface gave the results shown in Fig. 3. This chromatogram was generated from 100 ng of each analyte. At the 1-ng level, only terbufos gave a reasonable response.

To check for chromatographic peak tailing of high boiling analytes because of cold spots on the interface, lauryl laurate (dodecyl dodecanoate) was chosen. Lauryl laurate has been reported to be a useful compound for checking GC–chemical ionization MS performance[12]. The analysis used the same chromatographic conditions as those employed for terbufos and it oxidative metabolites with the exceptions of extending the temperature program to 220°C and raising the transfer line temperature to 200°C. The total ion current chromatogram from an 11-ng splitless injection is illustrated in Fig. 4 and shows no evidence of peak tailing. Comparable chromatographic performance was also obtained on the higher boiling *n*-hexatriacontane (*n*-$C_{36}H_{74}$, b.p. = 265°C at 1 Torr) after extending the column oven temperature program to 300°C and raising the transfer line temperature to 250°C. Under these experimental conditions, *n*-$C_{36}H_{74}$ eluted at 285°C.

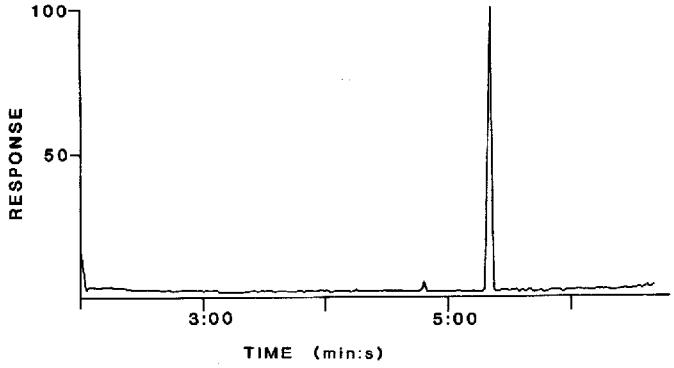

Fig. 4. Total ion current chromatogram from 11 ng of lauryl laurate.

In conclusion, a simple, direct GC–MS interface has been constructed for the Finnigan-MAT ion trap detector. The short, line-of-sight path permits the capillary GC column to be placed within the ion trap thus facilitating the analysis of labile analytes as shown for terbufos sulfoxide. The interface shows no evidence of chromatographic peak tailing due to active sites or cold spots. Capillary GC columns can be easily inserted and removed from the interface, and the interface can be readily constructed and retrofitted to existing ion trap detectors. While used here with short capillary columns for pesticide residue analysis, the interface is just as applicable for capillary GC–MS using much longer columns. Improved chromatographic performance could be achieved by operating at subambient inlet pressures[8,13], but with a resultant increase in the complexity of the GC–MS system.

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