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Large volume injection in gas chromatography using the through oven transfer adsorption desorption interface operating under vacuum



Álvaro Aragón^a, Rosa M. Toledano^a, Sara Gea^a, José M. Cortés^{a,*}, Ana M. Vázquez^b, Jesús Villén^a

^a Escuela Técnica Superior de Ingenieros Agrónomos, Universidad de Castilla-La Mancha, Campus Universitario s/n, 02071 Albacete, Spain

^b Facultad de Educación, Universidad de Castilla-La Mancha, Campus Universitario s/n, 02071 Albacete, Spain

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ABSTRACT

The present work describes a modification of the Through Oven Transfer Adsorption Desorption (TOTAD) interface, consisting of coupling a vacuum system to reduce the consumption of the helium needed to totally remove the eluent for large volume injection (LVI) in gas chromatography (GC).

Two different retention materials in the liner of the TOTAD interface were evaluated: Tenax TA, which was seen to be unsuitable for working under vacuum conditions, and polydimethylsiloxane (PDMS), which provided satisfactory repeatability as well as a good sensitivity. No variability was observed in the retention times in either case. Solutions containing organophosphorous pesticides in two different solvents, a polar (methanol/water) and a non-polar (hexane) solvent, were used to evaluate the modification.

The vacuum system coupled to the TOTAD interface allowed up to 90% helium to be saved without affecting the performance.

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1. Introduction

In the field of analytical chemistry, new methods are constantly being sought to reduce the quantity of sample used, simplify preparation of the sample and increase sensitivity, while minimizing the use of toxic solvents that are considered harmful to the environment. Most of the analytical methods that use gas chromatography (GC) involve previous sample preparation, which usually includes extraction and concentration steps. These steps, besides being time-consuming and needing large amounts of solvents, are the principal sources of error in the analytical process [1] hence the need for new methods that minimize these inconveniences.

Large volume injection (LVI) in GC and the direct coupling of liquid chromatography and gas chromatography (LC–GC) permit such sample preparation steps to be substantially reduced (less time and lower solvent consumption), while providing more reliable and sensitive results. LVI increases sensitivity and simplifies sample preparation since it avoids the extract concentration step where analytes loss are prone to occur [2], and even the need for an extraction step if large volumes of sample are injected

without prior preparation [3,4]. The direct coupling of LC–GC, besides permitting large volumes of sample or extract to be injected (the volume injected in LC is much higher than is normally injected in GC) leads to effective cleaning due to the great separation power of LC. For that reason, LC–GC coupling is suitable for the analysis of complex samples in which interferences must be eliminated before analysis by GC, while LVI is more suitable when the samples or extracts have a high degree of purity.

Whatever the case, the same difficulty is shared by LVI and the coupling of LC–GC: elimination of the large volumes of solvent – sample or extract solvents in the case of LVI and the LC eluent in the case of LC–GC – while retaining the analytes for transfer to the GC column. Several interfaces have been developed that permit the introduction of large volumes of sample, extract or LC eluent in GC. On-column interface, described by Grob [5] and later replaced by the Y-interface developed by Bierdermann and Grob in 2009 [6], and the loop-type interface are based on retention gap techniques [7]. On-column interface seem unsuitable for the LVI of polar solvents and for RPLC–GC because these eluents show poor wettability of the retention gap. A partial solution was proposed by Grob and Li [8], using an azeotropic mixture [7]. The loop-type interface does not require good wettability of the retention gap but its applicability is limited to high-boiling analytes [9]. Programmed Temperature Vaporizing (PTV) has become the most popular interface (described by Abel [10] and

* Corresponding author. Tel.: +34 967 599200x2507; fax: +34 967 599229.

E-mail address: josemanuel.cortes@uclm.es (J.M. Cortés).

developed by Vogt et al. [11,12]), the sample is injected into a liner placed inside a vaporizer. Several parameters must be optimized and the optimization process is time-consuming and tedious. PTV can also be used for the LVI of polar solvent and for RPLC–GC but only high boiling compounds can be analyzed.

Our research group was responsible for developing the Through Oven Transfer Adsorption Desorption (TOTAD) interface, first described in 1999 by Pérez et al. [13] and used for direct coupling of liquid and gas chromatography working in normal phase (NP) [14–16] and RP [17,18] in LC step and for LVI of sample or extract in the GC [19–21]. The TOTAD interface consists of a PTV injector, which has been substantially modified, with two electrovalves and a six port valve [22]. A retention material, usually Tenax TA, is placed inside the glass liner. Eliminating the solvent (carried out in a partial solvent evaporation mode) implies using more helium than is normally required in a GC operating conventionally, which represents a drawback because of the scarcity of helium reserves and its cost. Hence, the need to reduce the consumption of helium in three of the five steps involved in the TOTAD operation, where its consumption is particularly high: stabilization, transfer (or injection) and the remaining solvent elimination step [23].

Flores et al. [24] evaluated the use of adsorbents as retention material inside the liner for coupling LC–GC. With a PTV as interface and with the column connected and disconnected in each analysis, they compared the performance of the adsorbents with that of commonly used adsorbents. The adsorbents evaluated were polydimethylsiloxane (PDMS) and poly (50% phenyl–50% methylsiloxane) (OV-17), both on Volaspher A2, and the adsorbents were Tenax TA and Gaschrom. These authors concluded that the use of adsorbents, especially PDMS, was a good alternative since it presented advantages over the use of the most commonly used adsorbents. Subsequently, the same authors, Flores et al. [25] compared the performance of Tenax TA with that of the adsorbents PDMS and OV-17 as retention material inside the liner for pesticide residues in olive oils by direct coupling LC–GC. The obtained results showed that PDMS provided the best sensitivity and selectivity.

The aim of the present work was to substantially reduce the consumption of helium by carrying out the three above mentioned steps of TOTAD operation at a reduced pressure by connecting a vacuum pump to the interface. The vacuum system would favor solvent evaporation and so reduce the helium needed for

its elimination. System performance in vacuum conditions was evaluated by the LVI of standard solutions of pesticides in a polar and an apolar solvent.

2. Materials and methods

2.1. Materials

The organophosphorous pesticides used were diazinon, methylchlorpyrifos, fenitrothion, chlorpyrifos, parathion, phenthoate, chlorfenvinphos and ethion, all of which were supplied by Chem Service Inc. (West Chester, PA, SA). The methanol, water and hexane used as eluents were HPLC grade from Pestican (LabScan, Dublin, Ireland).

The retention materials used inside the glass liner of the TOTAD interface were Tenax TA, 80–100 mesh (Supelco, Madrid, Spain) as adsorbent material, and 50% (w/w) PDMS (Sigma-Aldrich, Madrid, Spain) in Volaspher A2 80–100mesh (Merck, Darmstadt, Germany), as adsorbent. These materials were selected because they involve two different retention mechanisms and they have previously been used with this interface. The glass liner of the TOTAD interface was packed with 1 cm of retention material between two plugs of glass wool to keep it in place. The retention material in the liner was conditioned under a helium stream, increasing the temperature by 50 °C 10 min⁻¹ to reach 300 °C, and maintained for 60 min at this final temperature.

Individual solutions of each of the pesticides were prepared in methanol or hexane at 1000 mg L⁻¹, and subsequently used to prepare solutions of 1 mg L⁻¹. Solutions were stored at 4 °C.

2.2. Instrumentation

The analyses were carried out using a 4000B Konik gas chromatograph with flame ionization detector (FID) equipped with a TOTAD interface (US patent 6,402,947 B1, exclusive rights assigned to KONIK-Tech, Sant Cugat del Vallés, Barcelona). A vacuum pump (KNF Neuberger GmbH, Laboport, Freiburg, Germany) was connected to the waste tubing (WT) (Fig. 1). A manual injection valve (model 7125 Rheodyne, CA) with a loop volume of 500 µL was used to inject the solutions. A ternary LC pump (model Konik 560) was used to push the high volume of solutions into the TOTAD interface. For data acquisition and processing the Konikrom 32 program (Konik, Sant Cugat del Vallés, Barcelona) was used.

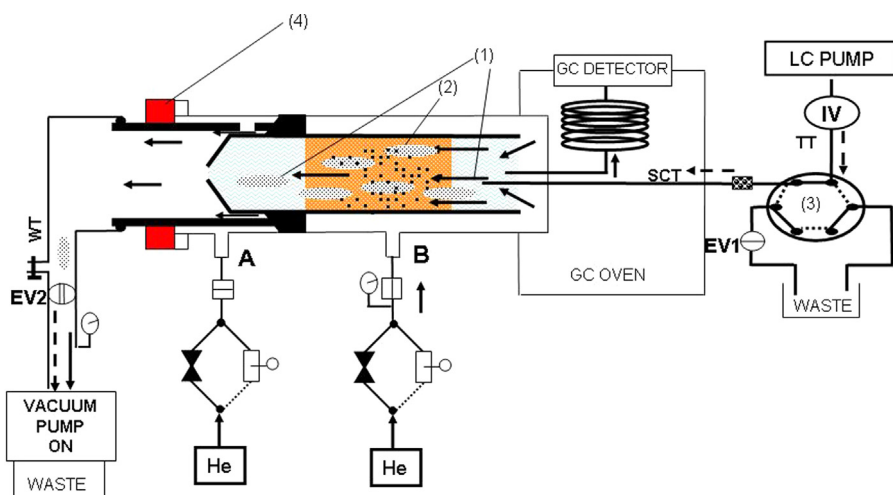


Fig. 1. Scheme of the TOTAD interface with vacuum pump coupled to WT during injection step. Symbols: (1) glass wool; (2) retention material; (3) six-port valve; (4) heated cover; (SCT) silica capillary tubing, 0.32 mm i.d.; (WT) waste tubing; (TT) transfer tubing; (IV) LC manual injection valve; (⊖) electro valve; (→) gas flow; (---) liquid flow; (⊞) pressure regulator; (⊞) filter; (⊞) needle valve; (⊞) restrictor; (⊞) opening-closing valve; (⊞) pressure gauge.

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