



Review article

Hand-portable liquid chromatographic instrumentation



Sonika Sharma^a, Luke T. Tolley^b, H. Dennis Tolley^c, Alex Plistil^d, Stanley D. Stearns^d, Milton L. Lee^{a,*}

^a Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, USA

^b Department of Chemistry, Southern Illinois University, Carbondale, IL 62901, USA

^c Department of Statistics, Brigham Young University, Provo, UT 84602, USA

^d VICI Valco Instruments, Houston, Texas 77055, USA

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ABSTRACT

Over the last four decades, liquid chromatography (LC) has experienced an evolution to smaller columns and particles, new stationary phases and low flow rate instrumentation. However, the development of person-portable LC has not followed, mainly due to difficulties encountered in miniaturizing pumps and detectors, and in reducing solvent consumption. The recent introduction of small, non-splitting pumping systems and UV-absorption detectors for use with capillary columns has finally provided miniaturized instrumentation suitable for high-performance hand-portable LC. Fully integrated microfabricated LC still remains a significant challenge. Ion chromatography (IC) has been successfully miniaturized and applied for field analysis; however, applications are mostly limited to inorganic and small organic ions. This review covers advancements that make possible more rapid expansion of portable forms of LC and IC.

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1. Introduction

The desire for immediate chemical analysis at the sampling point is one of the main driving forces for the development of hand-portable instruments. Analyzing samples at the point of collection rather than in the laboratory is useful in many applications, such as

space missions, forensics, homeland security, environmental contamination, geological studies, clinical analysis, and archeological studies, to name a few. This is especially important when health is at risk, such as in case of an oil spill or explosion, and in remote monitoring of locations contaminated with hazardous materials, such as biowarfare agents and/or toxic gases [1]. An ever-increasing number of harmful pollutants in water and air requires instruments that allow high throughput analysis as well [2]. Preservation of sample integrity is critical in most of these applications, and immediate

* Corresponding author.

E-mail address: milton.lee@byu.edu (M.L. Lee).

analysis after sampling would allow quick action to be taken during emergencies. With hand-portable instruments, both time and resources would be saved. However, the true potential of portable analytical instruments can only be realized if reduction in size does not compromise performance.

Optical instrumentation has dominated the portable analytical instrument market with the availability of portable Raman spectrometers [3,4], Fourier transform infrared spectrometers [4], near infrared spectrometers [4] and X-ray fluorescence analyzers [5,6]. These have been used in forensic analysis [7], space missions, organic and inorganic mineral analysis, stone and wall painting analysis, homeland security applications [8], and food quality control [9,10]. The popularity of optical instruments stems from their non-destructive nature and minimal sample preparation required before analysis. However, analysis of complex environmental and biological samples always requires separation [11], which is often performed using gas chromatography (GC), liquid chromatography (LC), or ion chromatography (IC), with GC and LC being the most versatile and popular techniques. Great effort has been made to miniaturize GC for field analysis, and several person-portable GC–MS systems are now commercially available [12–14]. GC is more amenable to portability than LC because of the use of gaseous mobile phases, which can be easily transported using compact pressurized gas cartridges weighing only a few grams; in some cases, air can be used directly as carrier gas. A GC operator does not have to worry about toxic waste disposal in the field, whereas portable LC demands transportation and handling of solvents, and disposal of solvent waste. Reducing the size and weight of LC pumps and bulky detectors has always been challenging. Therefore, limited work has been done in the past on miniaturization of LC [15–22].

There are two main challenges in making LC systems smaller: (1) minimizing solvent consumption and subsequent waste generation and (2) reducing size, weight and power requirements of the system. A split arrangement, both in mobile phase flow (pre-column) and sample injection, which is typically used with commercial capillary LC instruments, is unacceptable, considering the amount of waste generation and the loss of detection sensitivity when dealing with trace concentrations. Also, a chromatograph without a sensitive and compact detector would be of little value. Therefore, an LC system could only be qualified as portable if it fulfilled the following requirements:

- (1) weighs <7 kg and measures <1000 cubic inches (16,387 cm³);
- (2) contains all necessary electronics, digital interface and software integrated into a single unit;
- (3) is solar or battery-powered, allowing at least 8 h of operation
- (4) is easily operable with minimal supervision;
- (5) is rugged enough to withstand changes in temperature and humidity [1,23];
- (6) needs short instrument warm-up time;
- (7) uses low amounts of toxic organics [16] in compliance with green analytical chemistry principles [24];
- (8) is customized for capillary column use with a non-splitting flow arrangement, non-splitting injector, and low extra column volume to minimize dispersion;
- (9) is integrated with a small detector that has excellent sensitivity; and
- (10) is capable of binary gradient generation, competitive in performance to benchtop instruments.

Considering these requirements, it is obvious that some trade-offs must be made between portability and performance. If a portable LC could provide all of the advantages of a benchtop instrument in a compact size, current benchtop LC instruments would become obsolete. However, as long as portable LC can fulfill the requirements for field detection, or even provide preliminary

results on-site to complement further comprehensive analysis in a laboratory, it has its place. Moreover, in addition to environmental and point-of-care analysis, a portable system could also be utilized in a laboratory setting for routine analysis, reducing overall analysis costs, saving workspace and providing rapid results with minimal consumables.

The purpose of this review is to outline the main developments in miniaturization of LC instrumentation to allow the assembly of a person portable system. There are several LC reviews, which deal exclusively with micro- and nano-column chromatography and column advancements; however, none has dealt with system portability. This review initially focuses on miniaturization of LC components (column, pump, injector and detector) followed by a description of the construction and performance of the relatively few integrated portable LC systems. Finally, developments made in portable IC and microchip LC are described.

2. Development of portable LC components

2.1. Columns

LC instrument miniaturization has been strongly tied to column miniaturization. To reduce mobile phase consumption and required volume of sample, column dimensions have gradually decreased first to microcolumns (0.5–1.5 mm i.d.) and then to capillaries (0.01–0.5 mm i.d.). Capillary columns can reduce solvent consumption by 3 orders of magnitude as compared to conventional columns and, with resultant low sample dilution, detection sensitivity is better. However, as briefly described below, it took almost two decades of extensive research to fully exploit the true potential of capillary LC columns. Thus, early portable LC systems used conventional size packed columns due to their availability. However, the trend is changing and, with recent advancements in, and commercial availability of smaller and smaller particle-packed and monolithic columns, capillary columns are being incorporated in portable systems.

Packed microcapillary columns (50–200 μm i.d.) were first introduced by Tsuda and Novotny [25]. Long columns (several meters) packed with large diameter particles (>20 μm) became the only option for obtaining high chromatographic efficiencies at that time due to the lack of high performance small particles, high pressure pumps, and good column packing procedures [26,27]. Slurry packing of fused silica capillaries under high pressures markedly improved the homogeneity, efficiency and reproducibility of packed capillary columns [28–36]. Jorgenson's group reduced the internal diameter of packed capillary columns to 20 μm [35], and by the mid-90s, 12 μm i.d. capillary columns were reported [36]. These small i.d. columns could not be packed efficiently due to non-uniform particle sizes and shapes; however, a decrease in column diameter did improve column efficiency because of the plug-like flow obtained.

The trend of reducing particle size continued with sub-2 micron (1.5 and 1 μm) non-porous particles used for very high efficiency columns (>200,000 plates/m), leading to the development of ultra-high pressure liquid chromatography (UHPLC > 16,000 psi), as reported by Jorgenson's group [37,38]. Fast separations (few minutes) could be performed using high flow rates due to improved mass transfer characteristics with small diameter particles [39,40]. The first commercial UHPLC instrument appeared in 2004.

The chromatographic efficiency of packed capillary columns was remarkably improved recently by Wirth's group [41,42]. Colloidal crystal structures formed from sub-micron silica spheres (470 nm diameter) were prepared inside capillary columns, facilitating slip-flow. These columns gave more than a million plates per meter, which was at least 3.5 times higher than the plate numbers achieved with 1 μm nonporous particle packed columns. Plate

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