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Safety concerns in ultrahigh pressure capillary liquid chromatography using air-driven pumps

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Abstract

Ultrahigh pressure liquid chromatography (UHPLC) is an emerging technique which utilizes pressures higher than 10 000 p.s.i. to overcome the flow resistance imposed when using very small particles as packing materials in fused-silica capillary columns (1 p.s.i.=6894.76 Pa). This technique has demonstrated exceptionally high separation speeds and chromatographic efficiencies. However, safety is a concern when extremely high pressures are used. In this study, the safety aspects of capillary column rupture during operation were identified and carefully evaluated. First, liquid jets may be formed as a result of blow-out of the on-column frits or from rupture of the capillary at or near the column inlet. Second, incorrect installation of the capillary at the injector, failure of the ferrule used in the capillary connection, or rupture of the capillary can produce high speed projectiles of silica particles or column fragments. Experiments were carried out in the laboratory to produce liquid (water) jets and capillary projectiles using a UHPLC system, and the power density, an important parameter describing water jets in industrial practice, was calculated. Experimental results were in accordance with theoretical calculations. Both indicated that water jets and capillary projectiles under ultrahigh pressures might lead to skin penetration under limited conditions. The use of a plexiglass shroud to cover an initial length of the installed capillary column can eliminate any safety-related concerns about liquid jets or capillary projectiles.

Keywords: Ultrahigh-pressure liquid chromatography; Air-driven pumps; Safety concerns

1. Introduction

Theoretically, the best possible efficiency in the least amount of time for liquid chromatography (LC) is obtained when using very small diameter packing materials [1]. However, the minimum analysis time for a given separation is limited by the commercially available instrumentation because most conventional pumping systems have upper pressure limits of ~400 bar (6000 p.s.i.). Consequently, commercial columns packed with 1.5 μm nonporous particles are limited to approximately 33 mm in length. Such columns usually can separate samples within several minutes and produce 10 000–20 000 plates under typical operating conditions, ultimately resulting in faster separations with little or no gain in separation efficiency compared to conventional packed columns [2].

In 1997, MacNair et al. introduced ultrahigh-pressure liquid chromatography (UHPLC) in order to

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take advantage of the high efficiency potential of very small particles [3]. Subsequent studies have further demonstrated high efficiency and high speed in UHPLC [4–8]. Commercial pneumatic amplifier pumps (pressure controlled) used in these studies reached pressures as high as 5000 bar (72 000 p.s.i.). These pumps were used for packing columns as well as for chromatography. Tolley et al. recently reported the use of flow control in UHPLC [9]. Their system was based on a commercial pump which had been modified to operate at up to 1375 bar (20,000 p.s.i.). UHPLC requires some custom-made components or special modification of commercial instruments to withstand ultrahigh pressures. For example, laboratory-designed static-split injection valves were used for sample introduction because conventional injection valves could only endure pressures of 280–400 bar (4200–6000 p.s.i.). For safety reasons, MacNair and co-workers modified all original liquid seal components in their pump, and built a 3/16 in. thick steel box to enclose all of their UHPLC system components [3,5] (1 in.=2.54 cm). However, until now, little attention has been paid to carefully evaluating the safety concerns resulting from column rupture or failure in UHPLC during operation.

Column rupture in UHPLC can lead to two general safety concerns: liquid jets and capillary projectiles. In industry, high pressure liquid jets are used for a variety of purposes, such as manufacturing, cleaning, and dismantling [10]. In medical applications, liquid jets are used for cutting soft tissue [11,12]. However, high pressure liquid jet applications have also resulted in injuries because of their extremely high power densities [13–15]. A liquid jet could form in UHPLC if the capillary column broke or the on-column frits failed. Furthermore, other components, such as the injection valve, tubing, purge valve, pressure transducer and tubing connections, could wear out after being used for a period of time, which could also lead to formation of liquid jets. For example, to close a typical injection valve, a smooth cone-shaped surface of a needle tip is pressed into a channel orifice. The seal could fail after repeated use. It has been reported that the skin can be penetrated at a liquid jet power density of 1000–1500 W mm^{-2}, and bone is penetrated at a power density of 2200–3500 W mm^{-2} [16]. Incorrect installment of the capillary, breakage of the capillary or failure of the ferrule used in the capillary connector could lead to capillary projectiles being discharged in the liquid jet from the injection valve or tubing. The sharp capillary fragments represent the greatest potential risk of injury.

In this study, liquid jets and capillary projectiles caused by failures or ruptures in pressure-controlled UHPLC were investigated. Laboratory experiments were carried out to study the formation of liquid jets and capillary projectiles. Theoretical calculations were performed to estimate the power densities of liquid jets and the impact force of capillary projectiles under ultrahigh pressures. Modifications and practices to prevent any possible injury from ultrahigh-pressure liquid jets or capillary projectiles are suggested.

2. Experimental

2.1. Reagents

HPLC-grade water was obtained from Fisher Scientific (Fair Lawn, NJ, USA). Prior to use, the water was filtered through a 0.22-μm Durapore membrane filter (Millipore, Bedford, MA, USA) and degassed thoroughly. SFC-grade carbon dioxide and compressed nitrogen were purchased from Airgas (Salt Lake City, UT, USA). Fused-silica tubing was obtained from Polymicro Technologies (Phoenix, AZ, USA). The 1.0 and 1.5 μm nonporous isohexylsilane-modified (C_6) packing materials (Kovasil MS-H) were purchased from Chemie Uetikon (Uetikon, Switzerland).

2.2. UHPLC system

Details of the experimental apparatus and procedures used to perform UHPLC have been previously described [4]. Briefly, a double-head air-driven liquid pump (Model DSHF-302, Haskel, Burbank, CA, USA) with a piston area ratio (air drive area to liquid piston area) of 346 was used to generate the liquid pressure needed. The maximum air supply pressure was 150 p.s.i. (~10 bar), resulting in a pump pressure limit of 52 Kp.s.i. (~3600 bar). The internal volume of the pump was 4.5 ml. A cylinder containing compressed nitrogen was used to drive the pump. The outlet of this pump was connected to a three-way valve (Model 60-13HF2, High Pressure Equipment, Erie, PA, USA), one port of which was connected with the system pressure vent (two-way valve, Model 60-11HF2, HiP, Erie, PA, USA) and the other port with a high-pressure transducer via a tee (Model 60-23HF2, HiP). A short (5 cm) section of tubing was used for these connections. The high-pressure transducer allowed real-time monitoring of the column inlet pressure from 0 to 60 000 p.s.i. (0–4100 bar). A 13 cm section of tubing was used to connect one port of the tee with a laboratory-built injection block.

2.3. Measurement of the liquid jet velocity

Knowledge of the liquid jet velocity is necessary for calculation of the power density. Generally, the liquid jet velocity is hard to measure directly. In pressure-controlled UHPLC, if a liquid jet occurs, it can only last for a short time period until the pump reservoir becomes empty. The time between the start of liquid jet formation and complete pump volume expulsion was measured. The total volume of liquid from the jet was also collected and measured. The average liquid jet velocity could then be calculated from the total volume expelled divided by the product of expulsion time and jet cross-sectional area. The liquid used for measurement of the liquid jet velocity was water.

3. Results and discussion

3.1. Internal energy

The amount of stored energy in a system is a safety concern because too much stored energy may lead to an explosion or catastrophic failure. However, the actual amount of stored energy in the system is quite low, due to the low compressibility of solvents (i.e., water can be compressed approximately 10% at 40 000 p.s.i.). If air is compressed to 40 000 p.s.i., the stored energy is on the order of 10^8 J kg^{-1}, while for water at the same pressure, the stored energy is on the order of 10^5 J kg^{-1}, or three orders of magnitude smaller. Thus, from an energy standpoint, UHPLC is much safer than the high pressures would seem to indicate.

Nevertheless, ruptures or failures during operation in UHPLC are possible, and they can result in the formation of liquid jets and/or particle projectiles. Particle projectiles can represent the whole capillary with inlet end dislodged, capillary fragments and/or particles of packing materials. Details concerning formation and potential health risks of liquid jets and projectiles are discussed in the following sections.

3.2. Liquid jets

When the mobile phase is ejected out of a small diameter capillary or through an orifice under ultrahigh pressure, a liquid jet is formed. There are three main causes of liquid jets in UHPLC: first, breakage or dislodgement of the capillary column; second, blow-out of the on-column frits; and third, wearing out of system parts, such as the injection valve, tubing, purge valve, pressure transducer or tubing connections. In practice, liquid jets are rarely experienced from the third cause since these typically involve large volumetric flow-rates and the pump pressure decreases quickly (i.e., the pump can only maintain ultrahigh pressure when there is a low flow-rate). In addition, liquid jets are only remotely possible for well-packed columns from the second cause. Therefore, the main safety concern arises from liquid jets originating from breakage or dislodgement of the capillary column.

High-speed liquid jets can impose a cutting potential. Previous studies have shown that the skin is penetrated at a jet power density of 1000–1500 W mm^{-2} [16]. The power density can be calculated simply as the ratio of the hydraulic power and the area affected by the jet:

$$\text{Power density} = \left(\rho Q \cdot \frac{V^2}{2A}\right) \quad (1)$$

where Q is the volumetric flow-rate (ml s^{-1}), ρ is the fluid density (g ml^{-1}), A is the area affected by the jet (mm^2), and V is the average velocity of the liquid jet (m s^{-1}).

Calculation of the power density requires knowledge of the velocity or the volumetric flow-rate of the liquid jet, which is hard to measure directly. In pressure-controlled UHPLC, when a liquid jet occurs, it will last until the pump reservoir is empty. This pump reservoir drain time can be measured. The average volumetric flow-rates of various liquid jets were determined by dividing the total reservoir volume by the measured pump reservoir drain time. However, when ultrahigh pressures and short capillaries (capillaries remaining after breakage) are used, these drain times are hard to measure precisely due to the high speed flow-rates.

A simplified form of the mechanical energy equation for viscous flow through pipes can be used to describe the velocity of the liquid jet formed due to rupture in UHPLC:

$$V=\sqrt{\frac{2\Delta P}{\rho}\cdot\left(1+f\cdot\frac{L}{D}\right)} \quad (2)$$

where V is the exit average velocity from the broken capillary (m s^{-1}), ΔP is the pressure difference supplied by the pump (Pa), ρ is the fluid density (g m^{-3}), L is the length of capillary remaining (1–2 cm), D is the capillary inside diameter (cm), and f is the friction factor. Since f is dependent on V, the solution to Eq. (2) must be iterative. The empirical volumetric flow-rates obtained as described earlier were compared with those calculated from Eq. (2) (Table 1). It was found that the experimental results were in agreement with the theoretical calculations. Therefore, Eq. (2) was used for further prediction of liquid jet velocities at ultrahigh pressures.

When connecting the capillary to steel tubing or to the injector via vespel/graphite ferrules, the shortest feasible capillary length inside the injector is approximately 1 cm. Therefore, a worst-case scenario for liquid jet formation under our UHPLC conditions (i.e., remaining column length of 1.0 cm after breakage and a maximum operating pressure of 40 000 p.s.i.) was considered as shown in Fig. 1. Also, shown in Fig. 1 are the times required for the pump reservoir to empty.

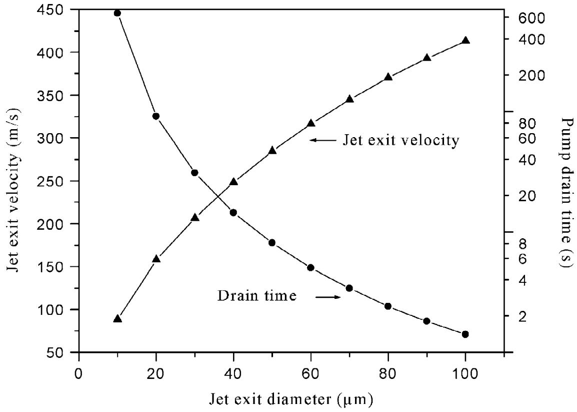

Fig. 1. Liquid jet exit velocity and time for pump cavity to empty vs. capillary diameter for a capillary length of 0.01 m and a pump pressure of 40 000 p.s.i.

At the capillary exit, the power density of the jet can be quite high as shown in Fig. 2. As the diameter of the jet exit orifice becomes smaller, the power density decreases because of the reduction in volumetric flow-rate. Unfortunately, the orifice diameter would have to be less than 5 μm to pose absolutely

Table 1
Comparison of experimental drain times and flow-rates with theoretical calculations[a]

Pressure (Kp.s.i.)	Experimental measurement		Theoretical calculation	
	Drain time (s)[b]	Flow-rate (ml s^{-1})[c]	Drain time (s)[b]	Flow-rate (ml s^{-1})[c]
6.00	25.2	0.163	25.2	0.153
9.00	19.7	0.198	20.3	0.192
15.0	14.4	0.271	15.2	0.256
21.0	11.7	0.332	12.6	0.310
25.0	10.6	0.369	11.4	0.342
35.0	–	–	9.5	0.413
40.0	–	–	8.8	0.445

[a] Assuming 2 cm capillary remaining after breakage.
[b] $n=4$.
[c] 4.1 ml pump reservoir volume.

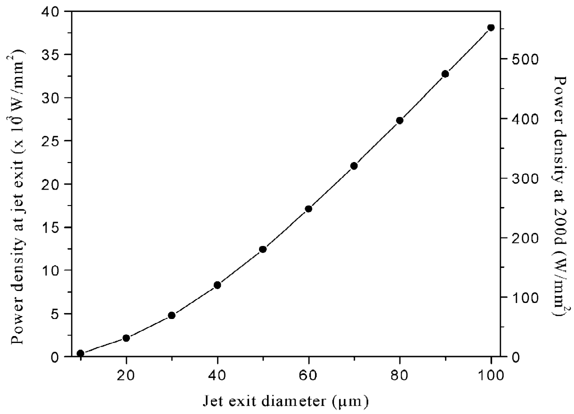

Fig. 2. Liquid power density at the jet exit and at 200*d* vs. *d* for a capillary length of 0.01 m and a pump pressure of 40 000 p.s.i.

no significant health threat. However, the jet velocity decreases with distance from the exit due to viscous interactions with the surrounding air; thus, the power density decreases (and the jet spreads). Also the jet tends to break up into droplets due to the surface tension of the liquid. Because of these effects, the damage potential is greatly reduced at even short distances from the jet exit. For example, a jet operating at a pressure of 3000 bar with a liquid jet diameter of 0.15 mm would cut plastic at a 10 mm stand-off, but would have no cutting power at a distance of 30 mm. Yanaida has shown that for liquid jets in air, at 200 diameters (200*d*) from the jet exit, the diameter of the jet increases to four times the exit diameter [17]. Thus, the velocity of the jet decreases to about 1/16th of the exit velocity. For capillaries of $d=10$ and 100 μm, this distance is 2 mm and 2 cm, respectively. Also shown in Fig. 2 is the liquid jet power density at a distance of 200*d* vs. the exit diameter. A safety factor of 2 was utilized in determining the power density; thus, the values are conservative.

Note in Fig. 2 that at the jet exit, the power density is larger than 1000–1500 W mm^{-2} for most diameters at 40,000 p.s.i. However, at 200*d* the power density is below this value for all cases. The breakup distance, i.e., the predicted distance where the jet breaks up into droplets, was calculated based on a correlation provided by Grant and Middleman [18]. The breakup distance under our conditions was at most 2.5 cm and, once breakup occurred, the danger of damage to soft tissue was eliminated. Thus, at distances greater than 2–2.5 cm (worst case), the danger due to the liquid jet was minimal.

The worst case for liquid jet formation was considered above. In fact, the risk decreases if rupture occurs further from the pressure inlet end of the capillary because the pressure drops gradually along the capillary length due to the resistance of the packed bed. Also, the densely packed particles in the capillary cause a delay in the formation of the liquid jet because it takes time to push the packing materials out of the capillary. This delay allows the operator to take some precautionary measures, such as turning off the pump.

3.3. Capillary projectiles

Capillary projectiles can be formed in UHPLC when the capillary end is discharged at high speed from the injector. This may be due to either breakage of the capillary, failure of the vespel/graphite ferrules, or incorrect installation of the capillary in the injector. Under this scenario, the existence of capillary projectiles in addition to the liquid jet is of concern. Incorrect installation of the capillary {not tightening the capillary connector in the injector (see Fig. 5 in Ref. [4] for details)} represents the worst case since the entire capillary may be ejected at high speed. This situation was created experimentally to produce projectiles and to study the effectiveness of various safety modifications (discussed in the next section). Since it was impractical to measure projectile speeds, theoretical calculations were used for further safety considerations.

If the capillary is completely removed, the size of the orifice through which liquid can be ejected is 360 μm (outer diameter of the capillary used), and viscous effects can be neglected so that the velocity can be computed from Eq. (2) where $f=0$. However, the pump cannot maintain a pressure of 40 000 p.s.i. with such a large flow-rate. In fact, under these conditions, the exiting jet velocity would be 166 m s^{-1}, with a corresponding pump pressure of 2000 p.s.i. For this jet velocity, the power density at the exit would be approximately 2300 W mm^{-2}; however, at 50*d* (1.8 cm) from the jet exit, the jet velocity is conservatively estimated to be approximately 83 m s^{-1} and the power density, 575 W mm^{-2}. Thus, beyond 1.8 cm, there is no danger from the liquid jet.

Propelled projectiles in the jet offer the greatest potential for injury. The worst case corresponds to a start up of the pump system with the capillary unrestrained. In this case, the capillary would accelerate out the end of the steel tubing or injector like a projectile. Assuming a long, straight projectile of diameter 360 μm, a force balance can be performed on the projectile to determine the velocity at which it would leave the tubing, i.e.:

$$\sum F = m \cdot \frac{\mathrm{d}v}{\mathrm{d}t} = m \cdot \frac{\mathrm{d}x}{\mathrm{d}t^2} \tag{3}$$

where v is the velocity (m s^{-1}) of the projectile and x is the position (m) of capillary projectile inside the tubing or injector. Also:

$$\sum F = PA = P\pi R^2 \tag{4}$$

and:

$$m = \rho_{\text{capillary}} \forall = \rho_{\text{capillary}} \pi R^2 L \tag{5}$$

where P is the pressure (Pa), A is the cross-sectional area (cm^2), R is the capillary outer radius (cm), $\rho_{\text{capillary}}$ is the capillary density (g ml^{-1}), ∀ is the volume (ml) of the fused-silica capillary, and L is the length (cm) of the capillary ejected.

Substituting Eqs. (4) and (5) into Eq. (3) gives:

$$P\pi R^2 = \rho_{\text{capillary}} \pi R^2 L \cdot \frac{\mathrm{d}x}{\mathrm{d}t^2} \tag{6}$$

Rearranging terms, the above equation can be written as:

$$\frac{\mathrm{d}x}{\mathrm{d}t^2} = \frac{P}{\rho_{\text{capillary}} L} \tag{7}$$

Solving Eq. (7), we obtain:

$$x = \frac{Pt^2}{\rho_{\text{capillary}} L^2} \tag{8}$$

When x is approximately equal to x_b, the initial length of capillary inside the tubing or injector, a force will no longer be applied. The time when this occurs is:

$$t = \left(\frac{\rho_{\text{capillary}} L^2 x_b}{P} \right)^{1/2} \tag{9}$$

From this time, the ejection velocity can be computed from:

$$\frac{\mathrm{d}x}{\mathrm{d}t} = v = \frac{P}{\rho_{\text{capillary}} L} \cdot t \tag{10}$$

Assuming a length of capillary in the tubing or the injector of 1 cm, the ejection velocity was modeled as a function of total capillary (projectile) length. The results are shown in Fig. 3. Although the pump cannot maintain a pressure of 40 000 p.s.i. long term because the actual pump pressure is limited by the volumetric flow-rate capability of the pump, due to liquid compression, the maximum pump pressure (40 000 p.s.i.) can be exerted on the capillary end initially. The maximum projectile velocity can be as great as 350 m s^{-1}, and this velocity represents an upper limit. However, the actual velocity will be much smaller because: (1) there is usually some restraining force on the capillary (it is not simply free to be ejected); (2) the capillary will not typically move as a straight, rigid whole when it is ejected, resulting in rapid deceleration; (3) the capillary will likely break since the initial length will be typically 15–30 cm, and the breakage energy will reduce the kinetic energy of the ejected capillary; and (4) there is a pressure drop along the capillary, which reduces the initial force on the projectile, depending on the point of breakage.

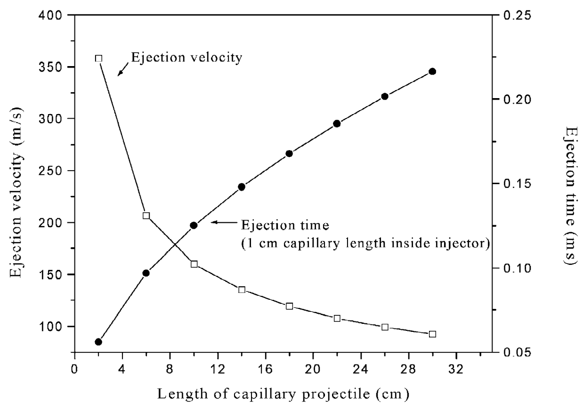

Fig. 3. Projectile ejection velocity and ejection time for a capillary length of 0.01 m inside the injector vs. capillary projectile length at a pump pressure of 40 000 p.s.i.

3.4. Specific recommendations

Modeling the ejection dynamics accurately is somewhat difficult because of their complex dynamic behavior. It can be said, however, that the maximum possible velocity of ejected projectiles from the steel tubing will be much less than 350 m s^{-1}. Even though the masses of the ejected capillaries would be small, ranging from 4 to 71 mg for 2–30 cm lengths, at these velocities, penetration of soft tissue would certainly occur, and damage to the eyes could be severe. This danger, however, can be eliminated by installing a plastic shroud over a short section of capillary column that extends out of the injector. There are two considerations regarding the required thickness of the plastic cover: penetration of the plastic, or impact and spalling on the exterior surface causing secondary projectiles. The maximum impact pressure of the ejected capillary is conservatively estimated as the product of the fused-silica density and the square of the maximum ejection velocity. For the worst possible case of an ejection velocity of 350 m s^{-1}, the maximum possible impact pressure will be less than 40 000 p.s.i. By comparing the impact force, $P\pi R^2$, with the force required to penetrate the cover, $\pi Rt\tau$, whether or not penetration is possible can be determined. In these equations, R is the radius (mm) of the projectile, t is the thickness (mm) of the plastic cover, and τ is the tensile strength (p.s.i.) of the plastic. Since $\pi Rt\tau$ is much greater than $P\pi R^2$ for plexiglass (approximately five times), for a thickness of 3.2 mm (1/8 in.) there is no danger of penetration. The fused-silica is brittle as compared to the ductile plexiglass, with a tensile strength (6900 p.s.i.) less than that of the plexiglass (9800 p.s.i.). Thus, the projectile will disintegrate at impact, resulting in a tremendous loss in energy and significantly decreasing the potential loading on the shroud. Thus, spalling cannot occur, and a plexiglass cover of 3.2 mm thickness is adequate for containment of any failure.

We have demonstrated such containment with a very simple setup as shown in Fig. 4. A plastic bottle with a small hole in the side was installed to contain the first several cm of the capillary. The capillary exited the plastic bottle at an almost right angle to the inlet capillary axis. The capillary was secured at the hole with epoxy. The inlet of the capillary was installed in the injector incorrectly (not tightening the capillary connector in the injector) so that the worst case scenario would result. As predicted, when the capillary was ejected, the loose end flew directly to the bottom of the inverted bottle and broke into pieces. The experiments were repeated several times (more than five) with an applied pressure of 40 000 p.s.i. It is worth mentioning that plastic bottles of different sizes were evaluated. The wall thickness of all of the plastic bottles was 1.5 mm which is in the safety range as discussed earlier. As long as the projectiles are contained, safety is assured. Another advantage of installing a shroud is that water jets are also contained.

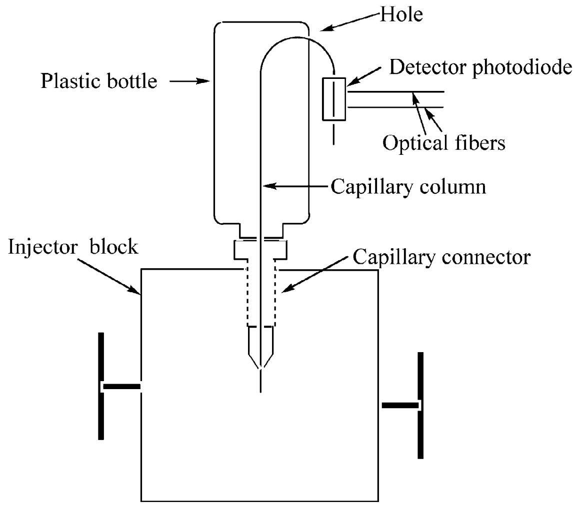

Fig. 4. Schematic of the UHPLC inlet system with safety modification.

4. Conclusions

Ruptures of columns and failures of the system components in UHPLC can lead to high speed liquid jets and capillary projectiles. Our analysis of liquid jets and capillary projectiles is based on the worst case. Even for the worst case, the power density of liquid jets at a distance of 200d, or approximately 2 cm, is lower than 1000–1500 W mm^{-2}. Capillary (particle) projectiles impose the greatest potential for injury. However, any possible injury can be eliminated by enclosing the first section of the capillary column in a V045I Plexiglass shroud.

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