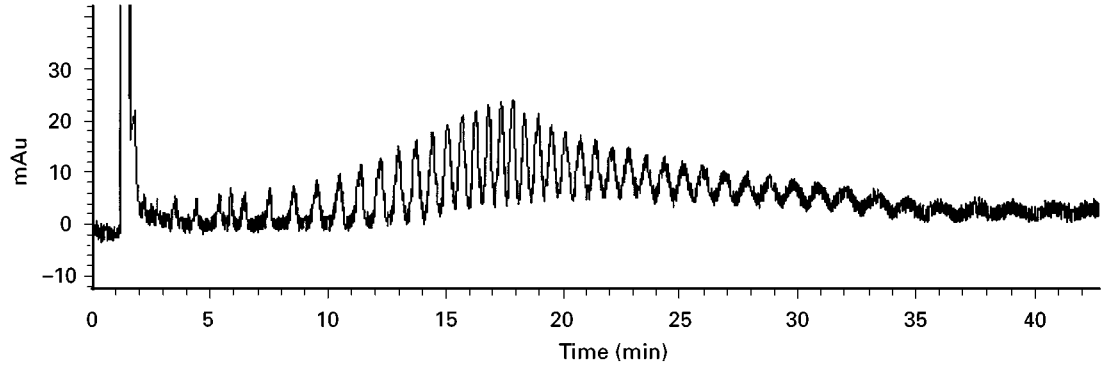

Figure 9 Chromatogram of the separation of a poly(methyl methacrylate).

The main disadvantages associated with using HPLC as a preparative separation technique are not experienced with SFC, namely the removal of the collected sample from the mobile phase and subsequent disposal of the mobile phase.

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PRESSURIZED FLUID EXTRACTION: NON-ENVIRONMENTAL APPLICATIONS

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Introduction

Pressurized fluid extraction (PFE), also referred to as accelerated solvent extraction (ASE), is a liquid

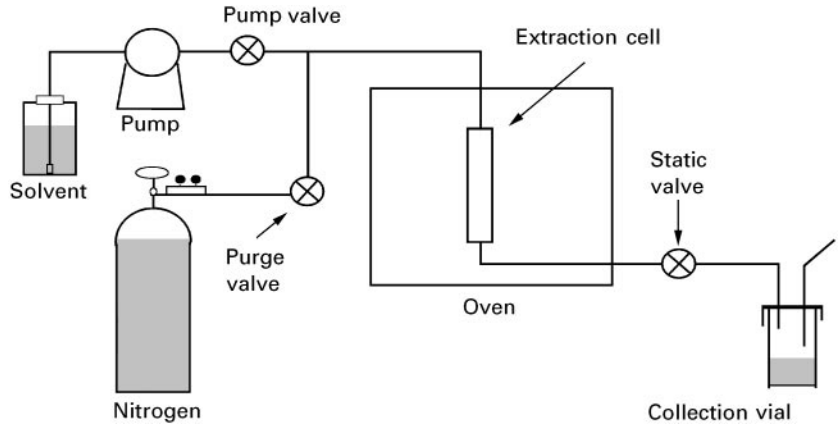

Figure 1 Schematic diagram of a pressurized fluid extraction (PFE) system.

solvent extraction technique developed and introduced by Dionex Corporation in 1995. While the initial applications focus of this technique was the environmental area, the versatility and ease of use of the approach has proven useful for laboratories performing extractions in the food and polymer industries, as well as in the pharmaceutical and consumer products areas.

Traditional reflux based extraction techniques such as Soxhlet extraction can take anywhere from 8 to 48 h to perform, with 24-h extractions common. Other liquid solvent based extraction techniques such as wrist shaker, hot-plate boiling and sonication require copious amounts of solvent and often involve labour-extensive steps such as filtering or concentration prior to extract analysis. One thing that they all have in common is operation at ambient pressure. An increase in temperature beyond the boiling point of the solvent is not possible owing to solvent evaporation.

Pressurized fluid extraction is performed by using the same solvents as in the traditional approaches, but at higher temperatures than is possible in these techniques. This increase in temperature improves the kinetics of the process, resulting in more efficient extractions (faster and using less solvent) compared with traditional approaches. The solvents are used under pressure so that their liquid state is maintained at the heated conditions. For example, solvents such as water, methanol, acetone or hexane are routinely used in PFE at 75–150°C. The solvents are maintained as liquids under pressure, normally at 10.4 MPa (1500 psi). PFE is therefore performed using very hot liquids to expedite the extraction process.

The flow-through design of the technique results in extracts which do not require the extended work of filtration as a means of separating the sample matrix from the extracted analytes. In further contrast to traditional extraction approaches, all of the basic steps of PFE are amenable to automation, freeing the analyst from the labour-intensive nature of most sample preparation protocols. Automated PFE systems can extract up to 24 sample cells, and have the necessary safety considerations for unattended operation built in.

Instrumentation

A schematic diagram of a PFE system is shown in **Figure 1**. The PFE extraction procedure consists of a combination of dynamic and static flow of solvent through a heated extraction cell containing the sample. These cells must be capable of safely withstanding the pressure requirements of the system, and are normally constructed of stainless steel, with frits in the end caps to allow the passage of solvent while retaining the solid sample. Disposable cellulose or glass fibre filters may be used in the cell outlets to avoid compaction of fine particles on the frit surface, which may impede the solvent flow. Solvent pressure is regulated via a high-performance liquid chromatography (HPLC) type pump and outlet valves control the flow of solvent from the cell to the collection vial. Compressed nitrogen is used to purge all of the liquid from the cell into the vial at the completion of the extraction.

Sample Preparation

Proper sample preparation is essential in order to obtain efficient and reproducible extractions. The ideal sample for extraction is a dry, finely divided solid, in which the extraction solvent can easily and thoroughly penetrate the sample matrix. Whatever can be done, within reason, to make samples ap-

proach this definition will be beneficial to the extraction process. Generally, samples should be prepared for PFE extraction in the same manner as traditional techniques. Samples with large particle size (> 1 mm) should be ground so as to increase the surface contact of the matrix and solvent. Wet or sticky samples should be mixed with drying agents such as sodium sulfate or Hydromatrix (pelleted diatomaceous earth), or with dispersing agents such as Ottawa sand prior to extraction. Typical sample sizes used in PFE are 1–30 g of solid or semi-solid material.

Sample Extraction Parameters

Extraction Solvent

As extraction parameters, solvent choice and temperature will have the greatest impact on extraction efficiency with PFE. An extraction solvent should be chosen which will solubilize the target analyte(s), but leave the majority of the sample matrix intact. This is normally done by matching the polarity of the solvent and analyte. PFE extraction can be performed with the entire range of aqueous and organic solvents, with the exception of strong mineral acids (hydrochloric, nitric, sulfuric) which will attack the stainless steel flow-path of the system. In those cases where an acidic pH is required, small amounts (1–5%) of acetic, phosphoric or other weak acids can be used. The choice of solvent should also be considered in the light of the post-extraction analysis technique. Solvents such as methanol and acetonitrile are suitable for direct HPLC injection, while solvents such as hexane, methylene chloride or acetone are more suitable for concentration and gas chromatography (GC) analysis. If the target compounds are easily oxidized, solvents should be degassed prior to use. It has been observed that solvents which perform only marginally well at ambient temperature often perform quite well at elevated temperature. This increases the range of solvent choices available to the analyst considering PFE, as more than one solvent may give good recoveries of target analytes. The selection of the appropriate solvent can then be made based on selectivity of extraction, solvent cost, safety and exposure factors, and compatibility with post-extraction processing steps. Solvent mixtures should also be considered in cases where minor adjustments to polarity are desired.

Extraction Temperature

PFE extraction can be performed from ambient temperature to 200°C. Increased temperature will increase the efficiency of the extraction process, and this should be optimized short of the point at which analyte degradation or excessive co-extraction of matrix components occurs. Many PFE applications are performed in the 75–150°C range, with 100°C as the recommended starting point for new methods development. In this temperature range, significant increases in extraction efficiency are observed without the breakdown of target compounds. If an extraction is to be performed on a compound with a known degradation point, then the PFE method should be developed to operate below that point. Extractions performed at low (40–70°C) or ambient temperatures may be sufficient for analytes which are weakly or only surface bound to the sample matrix. The extracts generated using PFE will be similar in composition to those produced by other techniques using the same solvents. If a post-extraction clean-up step is required following a Soxhlet extraction, the same process will most likely need to be performed following PFE.

Extraction Pressure

Although essential to the process, pressure is not generally considered a critical parameter in PFE. Normal operating pressures of 10.3–13.8 MPa (1500–2000 psi) are well above the threshold pressures required to maintain the solvents in their liquid states at PFE operating temperatures. The main purpose of using pressures in the ranges indicated is to provide rapid filling and flushing of the extraction cells. Typical PFE extractions are performed in 12–20 min, although this time can be extended for difficult samples. In addition, multiple static cycles can used periodically to introduce aliquots of fresh solvent during the extraction process.

Method Development

When developing a method for PFE the following approach has proven useful. A representative sample should be prepared as outlined above; select an extraction cell size which most closely matches the desired sample size. The extraction cells do not need to be filled completely, but a full cell will use less solvent in the extraction process than a partly filled one. Select the extraction solvent using the considerations listed above, although normally the same solvent or solvent mixture used in a traditional liquid extraction method is used. Extract the sample starting with the standard PFE conditions: pressure = 0.3 MPa (1500 psi), temperature = 100°C, heat time = 5 min, static time = 5 min, flush volume = 60% of cell volume, purge time = 60 s, static cycles = 1.

Extract the same sample multiple times in order to assess the efficiency of the method. If there is significant analyte present in the second or third extracts,

adjust the following parameters (one at a time), and repeat the validation process:

1. Increase the temperature (use 20°C steps).
2. Add a second or third static cycle.
3. Increase the static time (use 5-min increments).

If these steps do not result in a complete extraction, re-examine the sample preparation steps and/or the choice of extraction solvent.

Applications: Food

PFE extraction in the food industry is used for both the analysis of natural components such as fat, and to detect the presence of contaminants such as residual pesticides. New labelling requirements require food manufacturers to describe more accurately the total fat content of their products. This requires adequate monitoring of both raw and processed food samples. Pesticide residue analysis in foods has been, and will continue to be, a persistent and necessary analytical challenge. Proper selection of extraction solvent with food matrices can limit the high level of co-extractables typical with these sample types.

Fat Extraction

The determination of total fat in powdered infant formula is performed using a solvent mixture of hexane–acetone (4 : 1) at 100 or 125°C. Three 5-min static cycles are used in the method. Milk-based formulas are prepared by mixing 1 g of sample with 3 g of hydromatrix prior to cell loading and extraction at 125°C. Soy-based and hydrolysed milk-based formulas are mixed with wet hydromatrix (3 g + 0.4 g water) and extracted at 100°C. PFE extraction of these samples can be performed without the aggressive alkaline pretreatments required by some methods. Extraction results were compared directly with results obtained using alkaline pretreatment followed by Majonnier extraction with a mixture of petroleum ether, diethyl ether and ethanol (AOAC Method 932.06). The results obtained for the PFE extracts averaged 99.7% of the Mojonnier results for six different formula types, including a certified reference material (SRM 1846) available from the National Institute of Standards and Testing (NIST). The fat content was determined gravimetrically, and verified by fatty acid methyl ester (FAME) analysis.

Fat extraction from a variety of meat samples is performed by mixing 3–4 g of a homogenous meat sample with 6 g of hydromatrix. Moisture can be removed from the samples by drying in a microwave oven prior to extraction. Up to five samples can be dried at once in an 800 W oven at full power for 3 min. Samples are then extracted using either petroleum ether or hexane at 125°C, with two, 2-min static cycles. Extraction results were compared with a 4-hour soxhlet extraction with petroleum ether (AOAC Method 90.39). The results for a variety of samples are shown in **Table 1**. The PFE method used here was shown to be useful for both low and high fat meat samples and results in a considerable time savings compared with the traditional approach.

Table 1 Pressurized fluid extraction (PFE) and Soxhlet results for the extraction of fat from a variety of high fat processed meat samples

Sample	*% Fat by PFE*	*Standard deviation (n = 3)*	*% Fat by Soxhlet*
Beef	40.74	1.12	40.85
Pepperoni	42.66	0.28	43.15
Chorizo	27.98	0.22	27.84
Bacon	46.66	0.82	46.83
Sausage	33.80	0.28	33.54

Pesticide Residues

Using methods originally designed for soil samples, pesticide residues can be efficiently extracted from food samples including raw grains and fruits and vegetables. Wet samples should be mixed with sodium sulfate or hydromatrix prior to extraction. Extraction of organochlorine pesticides is performed using a solvent mixture of 10% acetone in hexane at 100°C. This solvent mixture limits the amount of co-extractables which are present in extracts produced with higher percentage acetone mixtures. Organophosphorus pesticides can be recovered with acetonitrile, ethyl acetate, methanol or acetone/methylene chloride mixtures, at temperatures ranging from 60 to 100°C. The more polar pesticides and herbicides, such as the sulfonyl ureas, can be efficiently extracted with a mixture of acetone and 0.1 M ammonium carbonate aqueous solution (20 : 80) at ambient temperature.

Applications: Polymers

PFE extraction in the polymer area has focused on additive analysis and general product structure characterization. For quantitative extraction of polymer matrices, samples should be ground prior to analysis. This can be accomplished with a liquid nitrogen grinder (cryo-grinder) as opposed to conventional laboratory grinders. Another major consideration in PFE extraction of polymers is the choice of extraction solvent. In other application areas the solvents used in existing methods are generally transferred and used in the PFE method. Traditional polymer methods,

however, swell and/or dissolve the sample matrix by boiling in a nonpolar solvent, followed by cooling and precipitation of the polymer. In PFE extraction, the goal is to separate the target additives of components from the sample matrix. The use of nonpolar solvents in this application will simply dissolve the entire sample and move it to the collection vial, or worse, it will precipitate in the transfer lines and plug the system flow path. The strategy developed for PFE extraction of polymers is to select a relatively polar solvent, which will solubilize the target analytes while leaving the majority of the sample matrix intact. Temperature is used to soften the matrix and a small amount of nonpolar solvent is added to increase penetration of the matrix. Cellulose thimbles are often used inside the PFE extraction cells to facilitate loading of the ground sample (normally 0.5–1.0 g) and prevent softened polymer from sticking to the sides of the vessel. Since small amounts of polymer matrix may be present in the extracts, samples are normally passed through a syringe filter prior to HPLC or GC analysis.

Additives from Polypropylene and Polyethylene

UV stabilizers, antioxidants and antislip agents are routinely added to polymer formulations to modify their properties for specific applications. Extraction and analysis of these compounds is essential in order to monitor that formulation levels are within specification. The antioxidant products (Irganox®, Irgafos® (Ciba Inc.), Erucamide, etc.) are a group of compounds commonly used in both low and high density polyethylene (LDPE and HDPE, respectively) preparations. Extraction of these compounds is performed at 140°C, using a solvent mixture of 2.5% cyclohexane in isopropyl alcohol. Three, 3-min static cycles are used to produce optimum results. Using these conditions, values equivalent to the results from reflux based extraction methods can be obtained. Chimassorb®944 (Ciba Inc.) is extracted from polypropylene (PP) using acetonitrile at 150°C. PFE can also be used to monitor the loss of Irganox 1076 which occurs after γ-irradiation.

Plasticizers in PVC

Traditional extraction of plasticizer content from polyvinyl chloride (PVC) is performed according to ASTM D 2124 recommendations. This method uses 120 mL petroleum ether in a 6-h Soxhlet extraction. The PFE method developed for this application uses petroleum ether at 100°C, with three, 1-min static cycles. The method does not require post-extraction filtering. GC analysis of the compounds extracted – dioctyl adipate (DOA), trioctyl phosphate (TOP), dioctyl phthalate (DOP) and trioctyl mellitate (TOTM) – showed average recovery of 101.7% relative to the reflux method (**Table 2**).

Table 2 Extraction of placticizers from polyvinyl chloride (PVC) by pressurized fluid extraction (PFE) and Soxhlet

Compound	*PFE %*	*Soxhlet %*
DOA	9.81	9.56
TOP	9.50	9.28
DOP	9.42	9.35
TOTM	9.17	9.05

Total Extractables from Styrene-butadiene Rubber

Styrene-butadiene rubber (SBR) is used in the manufacturing process of many consumer products including automobile tyres. The total extractable content of the rubber consists of oils and organic acids, and is usually measured gravimetrically. The PFE method developed for this application uses 2-propanol at 150°C, with three, 3-min static cycles. The results shown in **Table 3** indicate an average recovery of 99.9% relative to the target value.

Structure Characterization

The extraction of monomers and oligomers from formed polymers is used as an indicator to assess the completeness of the polymerization reaction. The extraction of monomer (caprolactam) from nylon-6 and oligomers (dimer, trimer) from 1,4-butylene terephthalate (PBT) was performed at 170°C with hexane–ethanol (60:40). Results indicate that recoveries equivalent to Soxhlet were obtained.

Applications: Pharmaceuticals

PFE extraction is used in the pharmaceutical industry both in the quality control of finished products and in the characterization of raw materials and product candidates. Extractions are normally performed on 1–10 g samples at temperatures ranging from ambient to 100°C, using polar solvents such as water,

Table 3 Total extractables from styrenebutadiene rubber (SBR) by pressurized fluid extraction (PFE)

Sample	*PFE %*	*Standard deviation (n = 3)*	*% Recovery*
1	32.66	0.17	100.2
2	32.77	0.04	100.5
3	33.89	0.19	100.1
4	34.44	0.31	98.9

methanol, ethanol and acetonitrile. Tablets, plants and other samples of large size should be ground prior to extraction.

Natural Products

The extraction of capsaicinoids from cayenne fruit, Hypericin from St John's Wort and alkaloids from goldenseal root is performed using ethanol, methanol or acetonitrile as extraction solvents at 100°C. St John's Wort extracts were analysed by UV/VIS absorbance at 516 nm following alumina cartridge clean-up to remove co-extracted chlorophylls. Hypericin content (measured as total dianthrones) was determined to be 0.44% with a %RSD = 4.1 ($n = 4$). This was consistent with a label claim of a minimum 0.3% hypericin. Goldenseal root was extracted according to the same conditions and analysed for total berberine content. HPLC analysis showed a total berberine content of 1.44% with a %RSD of 2.8 ($n = 4$). Water was also investigated as a potential solvent but was shown to extract too much of the samples matrix, which complicated the final analysis. In other studies, PFE extraction was compared with Soxhlet, reflux and steam distillation techniques for the extraction of St John's Wort, horse chestnut seed, milk thistle fruit, tumeric rhizome and thyme herb. Using methanol and temperatures ranging from 50 to 100°C, results comparable to or better than those obtained following USP method guidelines were obtained.

Animal Feeds

Extraction of an animal feed containing spiked levels of an anti-schizophrenic drug being tested in rats was performed at 100°C with methanol. Cattle feed containing the veterinary medicinal lasalocid (coccidiostat) was extracted using methanol containing 0.3% acetic acid at 80°C. Both extracts were analysed by HPLC and produced results comparable (96–105%) to the existing wrist-shaker techniques.

Transdermal Patches

Extraction of finished product is an essential part of the pharmaceutical quality control process. Transdermal patches containing nitroglycerin were extracted using ethanol at ambient temperature. The backing was peeled off of the patch and the sticky side was pressed onto sand in order to prevent the patch from sticking to itself. The patch, either 10 or 20 cm^2, was then curled into the extraction cell and Ottawa sand added as inert filler. The results shown in **Table 4** indicate a recovery of the active compound at 96–99% of the result obtained from the standard method, which involved extended shaking and extract filtering prior to analysis.

Table 4 Recovery of nitroglycerin from transdermal patches using pressurized fluid extraction (PFE) and wrist shaker methods

Sample	*PFE (mg)*	*SD (n = 10)*	*Wrist shaker (mg)*	*SD (n = 10)*
10 cm^2 patch	31.4	0.44	31.7	0.48
20 cm^2 patch	62.0	2.4	64.6	0.71

Ocular Inserts

Extraction of diclofenac sodium (anti-inflammatory) from an ocular insert used following cataract surgery was performed using methanol at 100°C . The polymeric tube containing the drug was cut into pieces prior to loading into the extraction cell. Results showed a recovery of 99.1% with a %RSD of 2.5 ($n = 8$). The PFE method replaces a 16-h Soxhlet extraction requiring 200 mL of solvent per sample.

Tablets

Extraction of felodipine (anti-hypertensive) in tablets was performed using acetonitrile at 100°C. Quantitative recovery (98%) from a single tablet was obtained by wrapping the tablet in a filter paper and crushing it. The crushed tablet and filter paper were then added to the extraction cell. Extraction of whole, uncrushed tablets resulted in less than adequate recovery.

Applications: Consumer Products

Tobacco

PFE extraction of tobacco is currently used to assess the total content of nicotine and other active and marker compounds, as well as flavour characterization and carbohydrate and sugar analysis. The extraction of raw and processed tobacco is performed with a variety of polarity solvents and at temperatures ranging from 50 to 100°C, depending on the types of compounds being targeted.

Detergents

Extraction of the organic constituents of granular and liquid detergents is performed using ethanol at 150°C. The conventional reflux extraction method used to determine total alcohol extractables is a multi-step process requiring 700 mL of solvent and 4 h to complete. The PFE extraction method for this sample is complete in 12–18 min and uses 15 mL of solvent per sample. Liquid detergent samples are mixed with an equal weight of hydromatrix prior to loading into the extraction cell. The results summarized in **Table 5** show an average recovery of

Table 5 Recovery of organic extractables from granular and liquid detergents using pressurized fluid extraction (PFE) and reflux extraction methods

Sample	*PFE %*	*Reflux %*
Granular 1	22.40	22.16
Granular 2	33.49	34.10
Granular 3	39.22	38.50
Granular 4	22.76	21.80
Granular 5	30.69	30.10
Liquid 1	45.35	44.35
Liquid 2	55.87	55.25

101.6% obtained with PFE compared with the conventional method, with a threefold improvement in reproducibility.

Textiles

The total extractable content of sized warp yarns is typically performed using AATCC Method 97-1995. This method is a series of three extractions using water, enzyme (bacterial amylase) and trichloroethane (TCE). Cotton and cotton/polyester yarns are extracted using PFE with water at 180°C, followed by extraction with TCE at 50°C (no enzyme was used). This method yields results slightly greater than those obtained with the standard method for total extractables by gravimetric determination and iodine spot test for polyvinyl alcohol and starch removal.

Paper

Total extractables from paper pulp is performed using methylene chloride at 125–150°C. Samples are prepared by cutting or shearing into small strips prior to cell loading. Three, 3-min static cycles are used to generate results of 101% of the standard reflux extraction method (analysis performed gravimetrically).

Summary

Conventional extraction times range from 4 to 48 h whereas PFE extractions are normally performed in 12–20 min. While the decrease in extraction time is favourable for most laboratories in general, it can be critical for those industries where laboratory data is used in feedback control of production cycles and manufacturing quality control. The volume of solvents used in PFE can be as much as 10–20 times less than traditional extraction methods. When factors such as safety and analyst exposure, as well as solvent purchase and disposal costs are considered, the benefits of PFE can be quite substantial for most laboratories. When compared directly with traditional extraction techniques, the recoveries generated by PFE normally equal or slightly exceed the comparative method. The ability of PFE to use the same liquid solvents used in traditional methods allows for rapid conversion to this technique, without much effort involved in methods development. Once a PFE method has been developed for a class of compounds, that same method can be successfully applied to a variety of matrix types without adjustment to extraction parameters. This lack of matrix dependency has allowed a very small set of standard methods to be applied to a large number of sample types.

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