Sains Malaysiana 45(2)(2016): 237–245

Troubleshooting and Maintenance of High-Performance Liquid Chromatography during Herbicide Analysis: An Overview

(Merumus Masalah dan Penyelenggaraan Kromatografi Cecair Berprestasi Tinggi semasa Analisis Herbisid: Suatu Gambaran Keseluruhan)

TAYEB, M.A.*, ISMAIL, B.S., KHAIRIATUL-MARDIANA, J. & GOH CHOO TA

ABSTRACT

Glufosinate ammonium or ammonium salt (ammonium-(2RS)-2-amino-4- (methylphosphinato) butyric acid; $C_5H_{15}N_2O_4P$) is a commonly used polar herbicide in Malaysia and present in a variety of environmental waters at the sub-ppb level. Thus, glufosinate ammonium is analyzed in soil and water using high-performance liquid chromatography (HPLC), which is a complex yet the most powerful analysis tool. HPLC is tremendously sensitive and highly automated and HPLC instrumentation and machinery have improved over the years. However, typical problems are still encountered. HPLC users and advanced learners require help in identifying, separating and correcting typical problems. All HPLC systems consist of similar basic components. Although it is a modular system, trouble can occur in each component and change the overall performance. Resolving these problems may be expensive. This review describes the different aspects of HPLC, particularly troubleshooting, common problems and easy guidelines for maintenance.

Keywords: Glufosinate ammonium; HPLC; maintenance; troubleshoot

ABSTRAK

Ammonium glufosinate atau garam ammonium (ammonium-(2RS)-2-amino-4-(methylphosphinato) asid butirik; $C_5H_{15}N_2O_4P$) adalah herbisid berkutub yang biasa digunakan di Malaysia dan hadir di dalam persekitaran air pada kepekatan sub-ppb. Oleh itu, ammonium glufosinate dianalisis di dalam tanah dan air dengan menggunakan kromatografi cecair berprestasi tinggi (HPLC) yang merupakan alat yang kompleks namun sangat jitu dalam menganalisis herbisid ini. HPLC sangat sensitif dan automatik serta prestasinya sering dipertingkatkan dari semasa ke semasa. Walau bagaimanapun, terdapat beberapa masalah sering dihadapi pengguna. Pengguna dan pengendali HPLC pada peringkat lebih tinggi memerlukan bantuan dalam proses pengenalpastian, pengasingan dan membetulkan permasalahan yang sering berlaku. Kesemua sistem HPLC terdiri daripada komponen asas yang sama. Walaupun ia adalah satu sistem yang modular, masalah boleh berlaku dalam setiap komponen dan mengubah keseluruhan prestasi. Menyelesaikan masalah ini mungkin memerlukan kos yang tinggi. Maka, ulasan ini menerangkan pelbagai aspek HPLC, terutamanya penyelesaian masalah HPLC, masalah yang sering berlaku dan garis panduan yang mudah untuk penyelenggaraan.

Kata kunci: Ammonium glufosinate; HPLC; merumus masalah; penyelenggaraan

INTRODUCTION

Glufosinate ammonium (ammonium-(2RS)-2-amino-4-(methylphosphinato) butyric acid; $C_5H_{15}N_2O_4P$) is a broad-spectrum herbicide. It is non-persistent in the environment because it is rapidly degraded in water, principally in the presence of light (Awis et al. 2013). High-performance liquid chromatography (HPLC) is the widely used analytical technique in herbicide separation for more than 35 years (Xiang et al. 2006). HPLC started in the 1960s as high-pressure liquid chromatography. By the end of the 1970s, the column materials and instrumentation of HPLC improved. HPLC boomed in the beginning of the 1980s. Since 2006, new terms for HPLC, such as ultra performance liquid chromatography (UPLC), rapid resolution liquid chromatography (RRLC), ultra performance liquid chromatography (UFLC) and rapid separation liquid chromatography (RSLC) popped up. In the early twentieth century, liquid chromatography was initially discovered as an analytical technique and was first used as a method of separating coloured compounds. The name 'chromatography' is derived from chroma, which means colour and graphy, which means writing. In 1906, Mikhali S. Tswett, a Russian botanist used a rudimentary form of plant pigments into pure constituents (Bliesner 2006). HPLC utilizes differences in the distribution of compounds into two phases: Stationary and mobile. The mobile phase designates the liquid that flows over the particles and the stationary phrase designates a thin layer created on the surface of fine particles. The solubility in the phases and the molecular size of each component in a sample contribute to different distribution equilibriums under a certain dynamic condition. Consequently, the components moved at different speeds over the stationary phases and are thereby separated from one another. The column, which is a

stainless steel tube, is packed with porous and superficially porous particles. The mobile phase is constantly fed into the column inlet at a constant rate by a liquid pump (Ravisankar et al. 2012). A sample injector is located near the column inlet and injects a sample. The injected sample enters the column with the mobile phase. The components in the sample migrated through it and passed to the stationary phase. The column compound migrates only when it is in the mobile phase. Therefore, compounds that tend to be distributed in the mobile phase migrate faster through the column, whereas those that tend to be distributed in the stationary phase migrate slower. In this manner, each component is separated in the column and sequentially elutes from the outlet. Each compound that elutes from the column is detected by a detector connected to the outlet of the column (Ismail et al. 2015d; Neue et al. 2001a; Tayeb et al. 2015b). A sampling valve with a loop is used to inject the sample in the following mobile phase just at the head of the separation column. In order to minimize unnecessary system peaks and to maintain good peak shapes, samples should be dissolved in a portion of the mobile phase. An in-line filter or guard column is utilized to prevent the contamination of the main column by small particulates (Tayeb et al. 2015a). A pressure gauge is inserted in front of the separation column to measure the column inlet pressure. The separation column contains the necessary packing to accomplish the desired HPLC separation.

Types of HPLC

The following types of HPLC are generally used in analysis and they generally depend on the phase system used in the process.

NP-HPLC

Normal phase chromatography (NP-HPLC) separates analytes based on polarity. Retention occurs through the interaction of the stationary phases of the polar surface with the polar parts of the sample molecules. From Figure 1 NP-HPLC uses a non-polar mobile phase, such as heptane, hexane, cyclohexane, dioxane, ethyl acetate and a polar stationary phase Sio_2. The polarity of a solvent depends on electronegativity differences. The electronegativity is equal i.e. (0) means the solvent is non-polar, if the difference >0 but <2 then it is polar. The analytes are non-ionic medium polar compounds and derivatives (Brandit & Kuppers 2002).

RP-HPLC OR RPC

Reversed phase chromatography (RP-HPLC) retention occurs through the partition of the analytes between the layer of the quasi-liquid stationary and the mobile phases. Figure 2 shows the non-polar stationary phase consists of n-octadecyl (RP-18), n-octyl (RP-8), ethyl (RP-2) or hydrophobic polymers and the polar mobile phase consists of methanol or acetonitrile/water or buffer. The RP-HPLC has polar aqueous mobile and non-polar stationary phases. Tetrahydrofuran and certainly dioxane are rarely used as additives in RPLC. Analytes are non-polar to medium polar compounds, such as hydrocarbons, alcohols, phenols, amines, carboxylic acids and derivatives with hydrophobic molecule parts and compounds with hetero-atoms (Ismail et al. 2013; Wiklund et al. 2005).

SEC

Size exclusion chromatography separates particles based on size. No interaction occurred between the analyte and

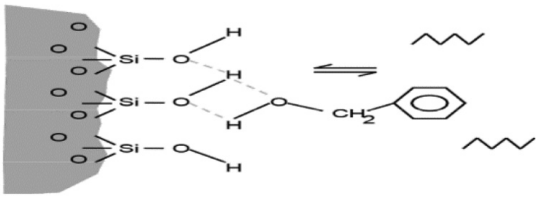

FIGURE 1. The mechanism of retention of the solute Ph-OH is shown in a typical normal phase chromatography separation. The charged form binds to the surface of the polar stationary phase and competes for the same positions with solvent molecules. If the polarity of the solvent is increased the more solvent molecules bind to the surface of the stationary phase and the solute (PhOH) elutes faster since it remains relatively untrained

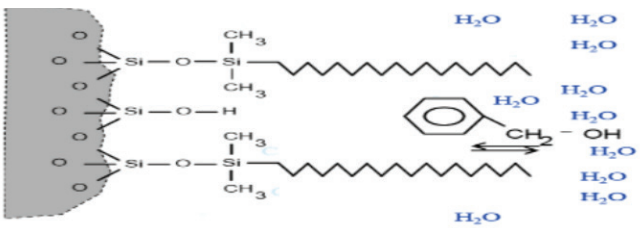

FIGURE 2. Reversed phase chromatography mechanism. The hydrophobic solute (nonpolar solute) binds to the surface of the hydrophobic (nonpolar) C-18 chain. A more polar mobile phase is used for solute elution to occur. The polarity of the mobile phase is decreased by changing its composition as elution progresses (gradient elution) to speed up the process

the gel surface. From Figure 3 large molecules elute faster and elution slowed down as the molecules became smaller (Cheng et al. 2000).

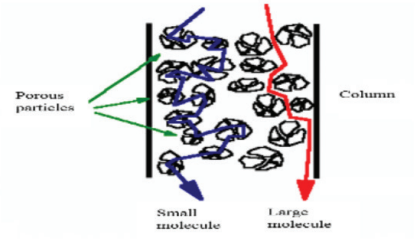

FIGURE 3. Size exclusion chromatography separation procedure

IEC

Ion exchange chromatography retention is based on the attraction between solute ions and charged sites bound to the stationary phase. Figure 4 shows cation exchange chromatography where positively charged molecules are attracted to a negatively charged solid support. Anion exchange chromatography negatively charged molecules are attracted to a positively charged solid support (Stavrianidi et al. 2005).

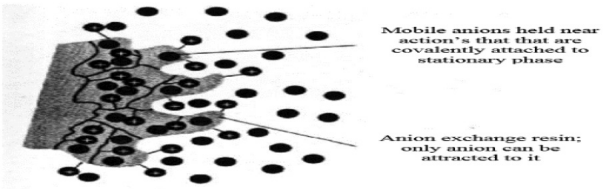

FIGURE 4. Ion exchange chromatography

INSTRUMENTATION

DELIVERY SYSTEM OF MOBILE PHASE

Mobile phases generally consist of water, aqueous buffer or mixtures of organic solvents with or without modifiers. The mobile phase must be delivered to the column over a wide range of flow rate and pressure. Ultra-sonication remove dissolved air and other gasses from the solvent (mobile phase). The capacity to generate a solvent gradient is another desirable feature in the solvent delivery system. Most of the time phosphate buffer (0.05 M) was used for glufosinate ammonium analysis, but it crystallized in the bottle and column capillaries, sometimes driving to an over-pressure of the pump and loss of analyte (Ismail et al. 2015c; Kirkland et al. 1998). Thus, phosphate buffer was replaced with phosphoric acid 0.2% because it adapted to the column and the column did not clog (Rathore 2003).

SEPARATION COLUMNS

The column is one of the important parts of an HPLC instrument. Figure 5 shows the columns are constructed of heavy wall or stainless steel to withstand high pressure (up to 6000 bar) and the chemical action of the mobile phase. Materials used for the construction of the connection tubing are stainless steel glass and peak polymer. The internal diameter of the analytical column is 1.0 to 4.6 mm, with lengths of 15 to 250 mm. Most of the columns ranged from 10 to 30 mm. For exclusion chromatography, columns are 50 to 100 mm long (Charde et al. 2013; Ismail et al. 2015a).

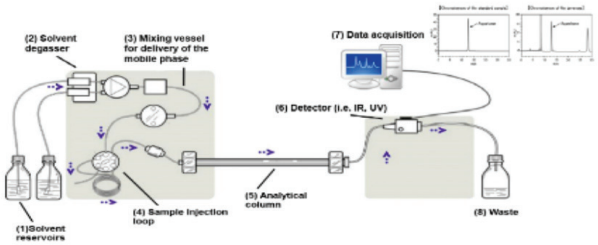

FIGURE 5. Liquid chromatography system

PORE SIZE

Pore size of the packing material represents the average size of the pores within each particle. The size of the molecules to be analyzed determines the pore size: MW < 3000 selects pore size of 60 to 100 Å; 3000 < MW < 10000 selects pore size of 100 to 130 Å; and 10000 < MW < 20000 selects pore size of 300 Å (Kaushal & Srivastava 2010).

SELECTION OF DETECTOR

The detector is a very important part of HPLC. Selecting the detector depends on the chemical nature of analytes, potential interference, limit of detection required, availability and/or cost of a detector. A sensitive universal detector for HPLC has not been devised yet. Thus, selecting a detector based on the problem is necessary (Iqbal et al. 2015).

UV DETECTOR

UV is an absorbance detector and provides good sensitivity for light-absorbing compounds. The UV absorbance difference in variable wavelength ranges for UV-VIS-190-900 and the suitable wavelength based on the compounds (Kima et al. 2015). Diagrammatic illustration of a UV-VIS detector optical system is shown in Figure 6.

VARIABLE WAVELENGTH DETECTOR

Figure 7 shows a variable wavelength detector with a relatively wide-band pass which offers a wide selection of ultraviolet (UV) and visible wavelength but at an increased cost than other detectors (Kwok et al. 2005).

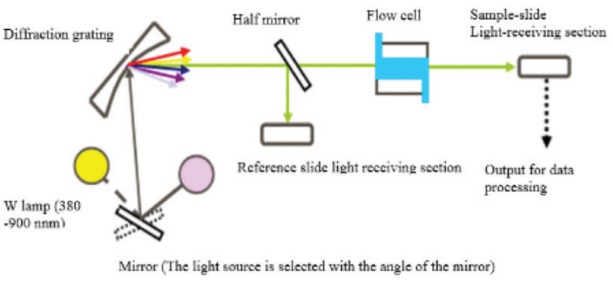

FIGURE 6. Diagrammatic illustration of a UV-VIS detector optical system

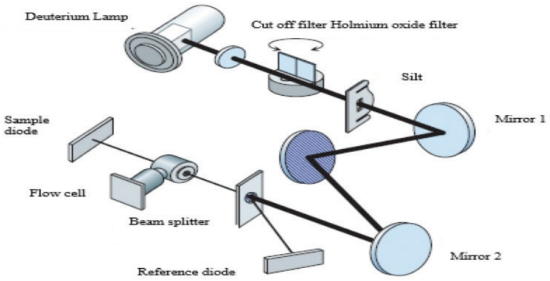

FIGURE 7. Diagrammatic illustration of a VWD detector optical system

FLUORESCENCE DETECTOR

Compared with UV-Vis detectors, fluorescence detectors offer a higher sensitivity and selectivity that allow for the quantification and identification of compounds and impurities in complex matrices at extremely low concentration levels (Cimadevillal et al. 2015). Substances can be determined using specific excitation and emission wavelengths. Fluorescence detection is suitable for trace analysis. Diagrammatic illustration of a FL detector optical system is as shown in Figure 8.

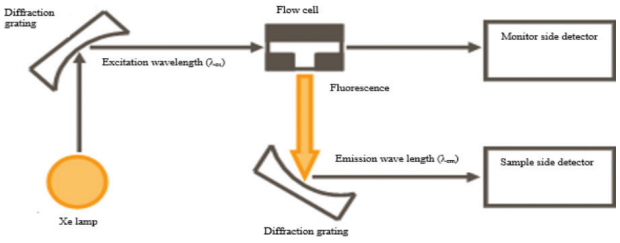

FIGURE 8. Diagrammatic illustration of a FL detector optical system

DIODE ARRAY DETECTOR

The photodiode array detector passes a wide spectrum of light though the sample and then the light is separated into individual wavelengths after passing through the sample. The spectrum of light is directed to an array of photosensitive diodes (Martin & Guiochon 2005). Figure 9 shows each diode can measure a different wavelength, which allows for the monitoring of many wavelengths at once.

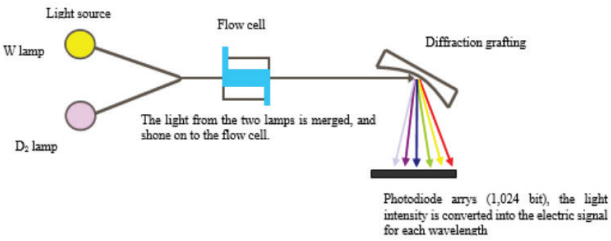

FIGURE 9. Diagrammatic illustration of a DAD detector optical system

MAINTENANCE OF HPLC

HPLC is a costly and sensitive technique. Consequently, HPLC equipment requires regular maintenance, thus, reducing the cost of routine maintenance is necessary. The equipment should be inspected weekly for leaks. A system suitability test is necessary prior to any analysis. Suitability test closely resembles the intended assay and should be performed to ensure that the system is operating within the established criteria. All hazards and safe handling practices should be familiarized before using the mobile phase solvents. The guidelines of the manufacturer regarding use, storage and disposal should be followed. The guidelines are normally provided in the material safety data sheets. Maintaining the HPLC storage condition is an important parameter. The system should not operate in a cold room or refrigerated area. The ambient temperature is 10 to 35°C and ambient relative humidity is 20 to 80% (Pieter et al. 2015). The parameters may vary depending on the manufacturers. A few basic assumptions to maintain the HPLC are as follows: The acetone should not be used as a solvent at 195 nm, the water should not be used as a gradient to hexane, methanol and water should not be mixed without degassing them, the solvent pH should not exceed 13 on a silica base column, but proper upper limit is pH=7, the system should be flushed with methanol or acetonitrile after running the buffer, the organic solvents should be filtered through the filter that is used for aqueos solution, the column frits should not be changed while pressure is still present, the pure cyclohexane should not be pumped above 2000 psi and the mobile phase container should not be tightly sealed. Finally, the instrument should be frequently calibrated using the appropriate procedures (Neue et al. 2001a; Ngwa 2010).

TROUBLE SHOOTING OF HPLC

Using a systematic approach is recommended in identifying any problems when troubleshooting the HPLC. The problems are categorized as baseline, chromatogram, pressure-related, leakage, and auto sampler problems.

Tables 1-6 and Figures 10-14; Tables 7-9 and Figure 15; and Tables 10-12 show and describe the possible causes and solutions of regular baseline noise, irregular baseline noise, baseline drift, split peaks, broad peaks, loss of resolution, smaller than expected peaks, no peaks, negative peaks, non-cyclic noise-fluid path problems, non-cyclic noise-detector electronics problems and cyclic noise-detector related problems and others, respectively.

TABLE 1. Regular baseline noise (Figure 10; Neng et al. 2015)

Possible cause	Solution
Leak	Check system for loose fitting, change pump seals if necessary, check pump for leaks, salt build-up, unusual noises
Air in mobile phase, detector cell or pump	Flush system to remove air from detector cell or pump, degas mobile phase
Temperature effect	Reduce differential or add head exchanger
Pump pulsations	Incorporate pulse dampener into system

TABLE 2. Irregular baseline noise (Figure 11; Mut et al. 2015)

Possible cause	Solution
Air bubbles in detector	Install back-pressure device after detector
Detector cell contaminated (even small amounts of contaminants can produce noise)	Clean cell by flushing with 1N HNO_3
Mobile phase mixture inadequate or malfunctioning	Repair or replace the mixture or mix off line if isocratic
Air trapped in system	Flush the system with viscous solvent isopropanol

TABLE 3. Baseline drift (Figure 12; Neue et al. 2001b)

Possible cause	Solution
Column temperature fluctuation	Use heat exchanger before detector, Control column and mobile phase temperature
Mobile phase recycled but detector not adjusted	Use new mobile phase when dynamic range of detector is exceeded, reset baseline
Gradient solvent B absorbs more than solvent A	Use base line subtraction, Try a new mobile phase
At high lab temperatures (28°C) more base line instabilities compared to lower lab temperatures (22°C) when using ACN/ water or buffer gradients and mixtures	Higher temperatures can enhance the polymerization of ACN resulting in building up of polymers. Filtration of ACN - eluent with empore SDB-XC polystyroldivinylbenzol filter

TABLE 4. Split peaks (Figure 13; Malviva et al. 2010)

Possible cause	Solution
Sample solvent incompatible with mobile phase	Inject sample in mobile phase or change the solvent whenever possible, inject samples in mobile phase
Contamination on guard or analytical column inlet	Change frit or replace column, use appropriate restoration procedure
Column blockage	Check the guard, in-line filter, column inlet and all associated tubing for blockage
Guard column voiding	Remove the defective guard and replace with a new one. Allow the system to reach equilibration and repeat the sample injection
Injection disrupting equilibrium	Dissolve the sample in mobile phase, use weaker diluent or make a smaller injection

TABLE 5. Broad peaks (Liu & Lee 2006)

Possible cause	Solution
Extra column effects: Recorder response time too high or tubing between column and detector too long or ID too large or detector response time or cell volume too large or column overloaded	Reduce response time or use as short a piece of 0.007-0.010 inch ID tubing as practical or use smaller cell or inject smaller column (e.g. 10 vs. 100 μL)
Peak represents two or more poorly resolved compounds	Change column type to improve separation
Column contaminated/worn out; low plate number	Replace new column with same type, flush old column with strong solvent if new column provides symmetrical peaks

TABLE 6. Loss of resolution (Figure 14; Juan & Tauler 2003)

Possible cause	Solution
Mobile phase contaminated	Prepare a new mobile phase
Obstructed guard or analytical column	Remove guard column and attempt

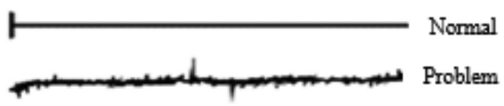

FIGURE 10. Regular baseline noise

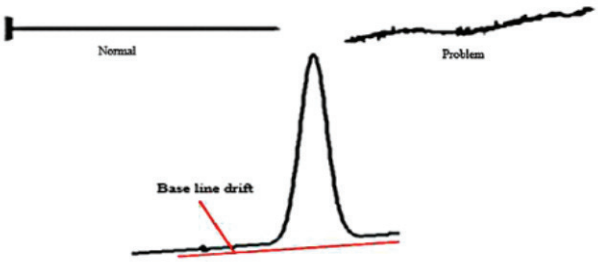

FIGURE 12. Baseline drift

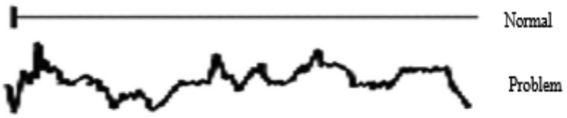

FIGURE 11. Irregular baseline noise

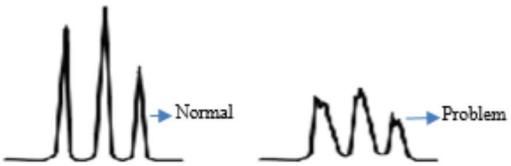

FIGURE 13. Split peaks

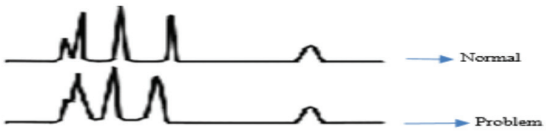

FIGURE 14. Loss of resolution

TABLE 7. Smaller than expected peaks (Kirkland & Henderson 1999)

Possible cause	Solution
Detector time constant too large	Use smaller time constant
Injection size too small	Use large sample loop
Detector attenuation too high	Reduce attenuation
Vial problem	Make sure that the vial sits correctly in the auto sampler and the needle is not obstructed when performing an injection

TABLE 8. No peaks (Leister et al. 2003)

Possible cause	Solution
No mobile phase flow	Start pump or check reservoir, loose fitting, salt build up, flush the system with methanol or isopropanol
Wrong mobile phase or wrong standard	Remove column and inject acetone solution to make a peak

TABLE 9. Negative peaks (Figure 15; Ismail et al. 2015b)

Possible cause	Solution
All peaks negative due to wrong polarity	Reverse leads or change detector polarity
Sample solvent and mobile phase differ greatly in composition (UV-detector)	Adjust or change sample solvent
Mobile phase more absorptive than sample components to UV web length	Use mobile phase that does not absorb chosen wavelength

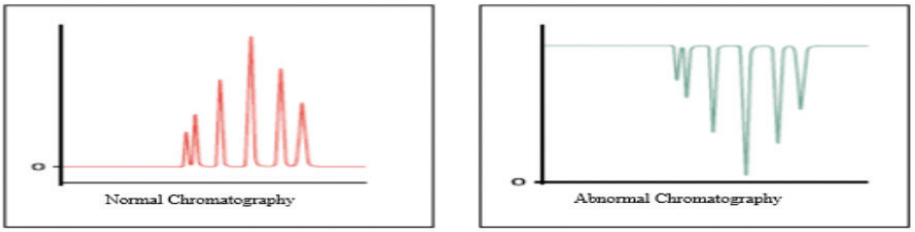

FIGURE 15. Negative peaks

TABLE 10. Non cyclic noise-fluid path problems (Gupta et al. 2015; Liu & Lee 2006)

Possible cause	Solution
Column contamination	Flush the column with mobile phase and monitor the baseline, if the baseline contains the same level of noise, even after changing the column than it indicates that the noise is due to another cause such as solvent miscibility, contaminated mobile phase or contaminated guards/in-line filter
Contaminated mobile phase	Clean all solvent inlet filters in a sonic bath with 6N HNO_3 and methanol
Electrochemical detectors only, air bubbles in reference electrode	Remove reference electrode from the instrument and gently shake it to dislodge the air bubble

TABLE 11. Non cyclic noise-detector electronics problems (Hongxia et al. 2004)

Possible cause	Solution
Detector not stable	The baseline will be stable once the detector is stabilized. After turning the detector on, allow it sufficient time for it to stabilized
Contaminated detector flow cell	Clean the detector flow cell cleaned with a 50/50 v/v mixture of THF/ water, than 100% THF if the system is used in normal phase
Reference electrode leak	ECD only-refer to the detector maintenance for repair

TABLE 12. Cyclic noise - detector related problems and others (Hassan et al. 2013; Ngwa 2010)

Possible cause	Solution
Long term detector temperature problems	The heater cycles on and off to maintain the detector temperature. Change the regularity of the on/off frequency to avoid baseline noise
Ambient temperature fluctuations	Stabilized the air temperature around this instrument and allow the system to return to equilibrium. If this is not possible replace the instrument to a laboratory position where the detector is thermally stable, avoid placing the instrument under direct sunlight

CONCLUSION

HPLC is probably the most universal type of analytical procedure. Its application areas include monitoring the pesticide level in the environment, glufosinate ammonium in soil and water level and analyzing the air and water pollutants, process control, forensic analysis, clinical testing and biochemistry research. HPLC also ranks as one of the most sensitive analytical procedures and is unique because it can easily separate multi-component mixtures. HPLC is made by a different complex component and it is hoped that this article helps in maintaining the HPLC system and avoiding common problems. The guidelines assist in reducing the maintenance cost and ameliorating the performance of the system. The entire review showed the common troubleshooting procedures for all the manufacturers.

ACKNOWLEDGEMENTS

This study was supported by research grant No. 06-01-02- SF 0827 & AP-2014-009 from Universiti Kebangsaan Malaysia and the Ministry of Education (MOE), Malaysia.

REFERENCES

Awis Sukarni Mohmad Sabere, Zuriati Zakaria & Ismail, B.S. 2013. Comparison of the level of organochlorine residues in paddy crops from two different cultivation practices. *Sains Malaysiana* 42(11): 1581-1584.

Bliesner, D. 2006. *Validating Chromatographic Methods*. New York: John Wiley & Sons, Inc. pp. 88-92.

Brandt, A. & Kueppers, S. 2002. Practical aspects of preparative HPLC in pharmaceutical development and production. *LC/GC North America* 21(1): 14-22.

Cheng, Y.F., Walter, T.H., Lu, Z., Iraneta, P., Grassi, B., Carmody, J.L. & Gara, R.P. 2000. Hybrid organic/inorganic particle technology: Breaking through traditional barriers of HPLC separations. *LC-GC* 18(11): 1162-1172.

Cimadevillal, H.M., Hevia, D., Miarl, A., Juan, C.M., Lombo, F. & Sainz, R.M. 2015. Development and validation of a single HPLC method for determination of α-tocopherol in cell culture and in human or mouse biological samples. *Biomedical Chromatography* 29(6): 843-852.

Charde, M.S., Kumar, J., Welankiwar, A.S. & Chakole, R.D. 2013. Review: Development of forced degradation studies of drugs. *International Journal of Advances in Pharmaceutics* 2(3): 34-39.

Gupta, V.K., Mergua, N., Kumawata, L.K. & Singha, A.K. 2015. A reversible fluorescence "off–on–off" sensor for sequential detection of aluminum and acetate/fluoride ions. *Talanta* 144: 80-89.

Hasan, N., Sher, N., Siddiqui, F.A., Ahmad, M., Shafi, N., Sial, A.A. & Baig, M.T. 2013. Novel HPLC method for quantitative determination of cefazolin sodium in pharmaceutical formulations. *Research and Reports in Medicinal Chemistry* 3(3): 21-28.

Hongxia, Y., Jing, C., Cui, Y., Shang, H., Ding, Z. & Jin, H. 2004. Application of toxicity identification evaluation procedures on wastewaters and sludge from a municipal sewage treatment works with industrial inputs. *Ecotoxicology and Environmental Safety* 57(3): 426-430.

Ismail, B.S., Mazlinda, M. & Tayeb, M.A. 2015a. The persistence of deltamethrin in Malaysian agricultural soils. *Sains Malaysiana* 44(1): 83-89.

Ismail, B.S., Prayitno, S. & Tayeb, M.A., 2015b. Contamination of rice field water with sulfonylurea and phenoxy herbicides in the Muda Irrigation Scheme, Kedah, Malaysia. *Environmental Monitoring and Assessment* 187(7): 406-419.

Ismail, B.S., Ooi, K.E. & Tayeb, M.A. 2015c. Laboratory assessment of 14c-Phenyl Metsulfuron-Methyl degradation in an oil palm plantation soil. *Journal of Oil Palm Research* 27(4): 403-416.

Ismail, B.S., Ooi, K.E. & Tayeb, M.A. 2015d. Degradation of Triazine-2-14C Metsulfuron–Methyl in soil from an oil palm plantation. *PLOS ONE* 10(10): 1-15.

Ismail, B.S., Mazlinda, M. & Tayeb, M.A. 2013. Adsorption, desorption and mobility of cypermethrin and deltamethrin in Malaysian soils. *International Journal of Plant, Animal and Environmental Sciences* 3(4): 23-29.

Iqbal, M., Bhat, M.A. & Shakeel, F. 2015. Development and validation of UHPLC-MS/MS assay for rapid determination of a carvone Schiff base of isoniazid (CSB-INH) in rat plasma: Application to pharmacokinetic study. *Biomedical Chromatography* 29(6): 876-882.

Juan, A.D. & Tauler, R. 2003. Chemo metrics applied to unravel multicomponent processes and mixtures revisiting latest trends in multivariate resolution. *Analytica Chimica Acta* 500: 195–210.

Kaushal, C. & Srivastava, B. 2010. A process of method development: A chromatographic approach. *Journal of Chemical and Pharmaceutical Research* 2(2): 519-545.

Kima, T.H., Kima, M.G., Shinb, B.S., Kimc, K.B., Leea, J.B., Paika, S.H. & Yooa, S.D. 2015.

Simultaneous determination of phenoxyethanol and its major metabolite, phenoxyacetic acid, in rat biological matrices by LC–MS/MS with polarity switching: Application to ADME studies. *Talanta* 144: 29-38.

Kirkland, J.J., Henderson, J.W., Martosella, J.D., Bidlingmeyer, B.A., Vasta-Russell, J. & Adams Jr. J.B. 1999. A highly stable Alkyl-Amide silica-based column packing for reversed-phase HPLC of polar and ionizable compounds. *LC-GC* 17: 634.

Kirkland, J.J., Van Straten, M.A. & Claessens, H.A. 1998. Reversed-phase high-performance liquid chromatography of basic compounds at pH 11 with silica-based column packings. *Journal Chromatography A* 797(1-2): 111–120.

Kwok, Y.C., Hsieh, D.P.H. & Wong, P.K. 2005. Toxicity identification evaluation (TIE) of pore water of contaminated marine sediments collected from Hong Kong waters. *Marin Pollution Bulletin* 51: 1085-1091.

Leister, W., Strauss, K., Wisnoski, D., Zhao, Z. & Lindsley, C. 2003. Development of a custom high-throughput preparative liquid chromatography/mass spectrometer platform for the preparative purification and analytical analysis of compound libraries. *Journal of Combinatorial Chemistry* 5: 322-329.

Liu, Y. & Lee, L. 2006. Ultrahigh pressure liquid chromatography using elevated temperature. *Journal of Chromatography* 1104(1-2): 198-202.

Martin, M. & Guiochon, G. 2005. Effects of high pressures in liquid chromatography. *Journal of Chromatography A* 7(1-2): 16-38.

Malviya, R., Bansal, V., Pal, O.P. & Sharma, P.K. 2010. High performance liquid chromatography: A short review. *Journal of Global Pharma Technology* 2(5): 22-26.

Mut, L., Grobosch, T., Domaß, T.B. & Frenzel, W. 2015. Toxicological screening of human plasma by on-line SPE-HPLC-DAD: Identification and quantification of basic drugs and metabolites. *Biomedical Chromatography* 29(6): 935-952.

Neng, N.R., Ahmad, S.M., Gaspar, H. & Nogueira, J.M.F. 2015. Determination of mitragynine in urine matrices by bar adsorptive microextraction and HPLC analysis. *Talanta* 144: 105-109.

Neue, U.D., Cheng, Y.F., Lu, Z., Alden, B.A., Iraneta, P.C., Phoebe, C.H. & Tran, K. 2001a. Properties of reversed phase packings with an embedded polar group. *Chromatographia* 54(3-4): 169-177.

Neue, U.D., Phoebe, C.H., Tran, K., Cheng, Y.F. & Lu, Z. 2001b. Dependence of reversed-phase retention of ionizable analytes on pH, concentration of organic solvent and silanol activity. *Journal of Chromatography A* 925(1-2): 49-67.

Ngwa, G. 2010. Forced degradation as an integral part of HPLC stability indicating method development. *Drug Delivery Technology* 10(5): 56-59.

Pieter, M.M., Kesel, D., Lambert, W.E. & Stove, C.P. 2015. An optimized and validated SPE-LC–MS/MS method for the determination of caffeine and paraxanthine in hair. *Talanta* 144: 62-70.

Rathore, A.S. 2003. Scale-up and optimization in preparative chromatography: Principles and biopharmaceutical applications. In *Chromatographic Science Vol. 88*, edited by Rathore, A. New York: Marcel Dekker.

Ravisankar, M., Sereya, K., Ananda Thangadurai, S. & Ramaiah, K. 2012. A review on: Preventive maintenance and troubleshooting of HPLC. *International Research Journal of Pharmacy*. www.Irjponline.com. Accessed on 20th August 2013.

Stavrianidi, A., Rodin, I., Braun, A., Stekolshchikova, E. & Shpigun, O. 2015. Single-run HPLC/ESI-LITMS profiling of ginsenosides in plant extracts and ginseng based products. *Biomedical Chromatography* 29(6): 853-859.

Tayeb, M.A., Ismail, B.S. & Khairiatul Mardiana, J. 2015a. Comparison of four different solid phase extraction cartridges for sample clean-up in the analysis of glufosinate ammonium from aqueous samples. *International Journal of Chem. Tech Research* 7(6): 2612-2619.

Tayeb, M.A., Ismail, B.S., Mardiana-Jansar, K. & Goh Choo Ta. 2015b. Glufosinate ammonium clean-up procedure from water samples using SPE. *AIP conference proceedings* 1678: 060024(1-7).

Wiklund, A.E., Dag, B. & Brita, S. 2005. Toxicity evaluation by using intact sediments and sediment extracts. *Marine Pollution Bulletin* 50(6): 660-667.

Xiang, Y., Liu, Y. & Lee, M.L. 2006. Ultrahigh pressure liquid chromatography using elevated temperature. *Journal of Chromatography A* 1104(1-2): 198-202.

Tayeb, M.A.*, Ismail, B.S. & Khairiatul-Mardiana, J.
School of Environmental and Natural Resource Sciences
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43600 Bangi, Selangor Darul Ehsan
Malaysia

Goh Choo Ta
Institute for Environment and Development (LESTARI)
Universiti Kebangsaan Malaysia
43600 Bangi, Selangor Darul Ehsan
Malaysia

*Corresponding author; email: atayeb91@yahoo.com

Received: 27 August 2014
Accepted: 5 August 2015