

## Simultaneous Determination of Inorganic Ions in Post-Blast Residues using Capillary Electrophoresis

Umi Kalthom Ahmad<sup>a\*</sup>, Nor Laili-Azua Jamari<sup>a</sup>, and Sivabalan A/L Nagayah<sup>b</sup>

<sup>a</sup>*Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor Darul Ta'zim*

<sup>b</sup>*Criminalistics Section, Department of Chemistry Malaysia, Jalan Sultan, 46661 Petaling Jaya, Selangor Darul Ehsan*

**ABSTRACT:** Capillary electrophoresis method for simultaneous determination of inorganic anions and cations was developed using 2,6-pyridinedicarboxylic acid (PDC) as a background electrolyte (BGE) with indirect UV detection. In order to detect both anions and cations simultaneously, the electromagnetic flow was reversed by the addition of cetyltrimethylammonium hydroxide (CTAH). The parameters which influenced the separation of inorganic ions such as electrolyte pH, concentration of PDC, applied voltage and temperature were investigated. Four anions ( $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{SCN}^-$ ) and three cations ( $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ) were successfully determined simultaneously in less than 7 min under the optimized conditions (25mM PDC, 0.5mM CTAH, pH 4.7). The method was applied to the analysis of post-blast explosive residues of black powder and ammonium nitrate-fuel oil (ANFO). The analytical performances of the method are discussed in terms of analysis time, repeatability, reproducibility, linearity of response and detection limits.

Keywords: Anions, cations, post-blast residue, capillary electrophoresis

### Introduction

Post-blast residues, particularly inorganic residues refer to the debris and inorganic residue left at a crime scene from the detonation of explosive. Homemade explosive device used by terrorists are normally based on inorganic salts and/or peroxides since these ingredients are readily available, low cost and can be purchased legally [1]. The major components in explosives are oxidizers such as nitrate, chlorate, or perchlorate; and fuel such as charcoal, sugar, or metal oxides. After detonation, explosive leave residues that contain a variety of components such as ionic species which are produced from the explosive reaction, with unreacted components of the original explosive mixture [2].

Determination of the level of inorganic anions and cations present in residue demands the identification of the original explosive [1]. Most of the chemicals species present in post-blast residues of low explosives are anions and cations. Examples of inorganic residue in most of the explosives are nitrite, nitrate, sulfate, chlorate, carbonate, perchlorate, ammonium, potassium and sodium [3-5]. The pattern of ionic components of post-blast residue often reflects the original composition, type, source and manufacturer of the explosive [6, 7].

Ion Chromatography (IC) has been the method of choice for the analysis of anions in post-blast residues [8]. Capillary electrophoresis (CE) is an

attractive alternative for post-blast residue analysis because of its versatility. It can be applied to detect inorganic ions, organic molecules and large biomolecules using the same instrument and same capillary in most cases, given that the composition of running buffer is changed. This method is also environmental-friendly since the resultant waste contains only small amount of organic modifier and the buffer can be discarded without causing any danger to the environment [9]. CE can also yield a high speed and high resolution separation on small amount of sample [10]. Its availability for different modes of separation and its possibility in interfacing with different detection system makes CE widely applicable in various fields [9].

Recently, several CE methods for simultaneous analysis of cations and anions have been reported [5, 11]. Padaraukas *et al.* [11] proposed a unique technique based on electromigration sample introduction from both end of capillary using indirect UV detection with two background electrolyte (BGE). After applying voltage, anions and cations migrated in opposite direction from different capillary end and ions were detected at the detector which placed on the middle of the capillary. They successfully separated four anions and five cations in less than 5 minutes.

Hopper and coworkers [5] described the separation of inorganic ion using single injection with indirect UV detection. Single BGE was used with addition of

anion and cation chromophore, cation selectivity modifier and organic modifier. Eight anions and five cations were successfully separated with a total run of 7 minutes.

In this study, a new CE method for simultaneous analysis of anions and cations by using 2,6-pyridinedicarboxylic acid (2,6-PDC) as BGE was developed. The method was optimized and then applied for the simultaneous determination of inorganic ions in post-blast residues of black powder and ammonium nitrate-fuel oil (ANFO).

## Experimental

### Chemicals

Inorganic anion and cation standards were prepared from their salt. The chemicals used were of analytical or reagent grade. It included calcium chloride from QRec (Malaysia), ferrous (II) sulfate, ferric (III) nitrate, and potassium thiocyanate from GCE laboratory (USA). For electrolyte preparation, the chemicals used were 2,6-pyridine dicarboxylic acid from Fluka Chemica (Switzerland) and cetyltrimethylammonium hydroxide solution (25% in methanol) from Sigma-Aldrich (USA). For pH adjustment of buffer, sodium hydroxide solution was prepared from sodium hydroxide pellets from QRec (Malaysia). All electrolytes and standards were prepared using 18.2 M $\Omega$  double distilled deionised water (DDDW) from Milipore ultrapure water system (Merck, Darmstadt, Germany).

### Instrumentation

CE instrument used in this study was an Agilent G1600 Capillary Electrophoresis System with indirect UV detection. For simultaneously separation of both anions and cations, the capillary used was a fused silica capillary with dimensions of 52 cm length x 50  $\mu$ m I.D. CE ChemStation software was used for system control, data collection and data analysis.

### Electrophoretic Procedure

The electrolyte was prepared containing 0.5 mM cetyltrimethylammonium hydroxide (CTAH) to reverse the direction of electroosmotic flow (EOF) [12] and BGE at a concentration of 25 mM. The pH was adjusted to 4.7 with 1 M NaOH. Prior to use, the capillary was pretreated with the following procedures: deionised water for 10 minutes, 0.1 M NaOH for 10 minutes and deionised water for 10 minutes. The capillary was pre-conditioned with 2,6-PDC buffer for 4 minutes for each run. Samples were injected in the hydrodynamic mode for 6 seconds. The capillary temperature was held at 15°C and the applied voltage was at -20 kV. Detection was carried out using UV diode array detector and the signal wavelength was set at 350 nm with reference at 230 nm.

### Preparation of Standards

All standards ( $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{SCN}^-$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ) were initially prepared at a concentration of 1000 ppm and diluted to a working concentration of 100 ppm. The solutions were sonicated for 5 minutes and filtered through 0.45  $\mu$ m membrane filter disc. A series of standard mixture solution ranging from 20 ppm to 100 ppm were produced which were used for the preparation of calibration curve. The LODs of each ion were evaluated by diluting 20 ppm standard mixture with DDDW and analyzed using the developed CE method. The lowest detectable concentration for each ion was based on signal-to-noise ratio of 2:1.

### Sampling

Samples of post-blast black powder residues were obtained from a previous sampling exercise [18] at Rompin, Pahang. Three fragments (BP-1, BP-2 and BP-3) were collected as post-blast residues of different explosive charge of 100 g, 150 g and 200 g, respectively.

For ANFO residues sample, a homemade bomb was made by police personnel at the sampling site using 30 g of ammonium nitrate mixed with diesel fuel oil that was filled into a plastic mineral bottle. A detonating cord containing PETN as a booster was attached to the plastic bottle. The homemade bomb was placed in the middle of a field and two different types of cloths (cotton and nylon) were placed 2.5 meters away from the bomb in a position 180° from each other. The cloths (23 cm x 32 cm) were tied to a metal plate fixed onto a small iron rod. After detonation, each cloth was collected and packed in snap-seal plastic bag.

### Sample Preparation

#### *i. Sample Preparation of Black Powder Residue*

For swabbing of black powder residue from bomb fragments, the cotton balls were pre-cleaned before use. The cotton balls were first cleaned by soaking them in DDDW for 15 minutes. The cotton balls were taken out and excess water squeezed out. The cotton balls were left to air dry for about 20 minutes. The pre-cleaned cotton balls were picked up using forceps and moistened with DDDW. The moist cotton ball was swabbed gently onto the surface of post-blast fragments to collect the residue.

Cotton swab containing the residue was placed in a 100 mL sample bottle and filled with 20 mL of DDDW. The sample bottle was sonicated for 10 minutes to extract the inorganic ions. After sonication, the cotton swab was removed from the sample bottle and the resultant solution was filtered through a cleaned 0.45  $\mu$ m membrane filter disc. The extract was diluted to 100 mL, followed by degassing for 5 minutes. The extract solution was placed in a

sample bottle and kept in a refrigerator prior to analysis.

#### ii. Sample Preparation of ANFO Residue

For ANFO residue deposited on cloth, the cloth was cut into smaller pieces and placed in a 50 mL sample bottle. Twenty mL of DDDW was added into the sample bottle followed by sonication for 10 minutes in order to extract the inorganic ions. The cloth pieces were removed from the sample bottle and the resultant solution was filtered through 0.45  $\mu\text{m}$  membrane filter disc and diluted to a total volume of 25 mL. The extract solutions were degassed for another 10 minutes and kept in the refrigerator prior to analysis.

#### Analysis of ANFO Control Sample

ANFO control sample used in the study was provided by the Royal Malaysia Police. A 0.01 g of ANFO control sample was dissolved in 10 mL volumetric flask using DDDW. The mixture was sonicated and filtered through a 0.45  $\mu\text{m}$  membrane filter disc. A 100x dilution was carried out by dissolving 1 mL of ANFO solution in 100 mL volumetric flask using DDDW. The diluted standard solution was kept under refrigeration prior to analysis.

## Results and Discussion

### Choice of BGE

For simultaneous separation of anions and cations, an indirect UV detection was utilized. This detection mode required that the electrolyte contained both cationic and anionic visualization agents. In this study, 2,6-PDC acid was chosen as BGE due to its

ability to form negatively charged complexes to permit indirect UV detection. 2,6-PDC is also known to have high sensitivity, high stability and UV responsive for metal cations [13, 14]. Formation of anionic complexes was also found to be highly dependent upon electrolyte pH and concentration of 2,6-PDC acid [15].

Once 2,6-PDC acid reacted with cations to form anionic complexes, they migrated toward positive electrode and allow for indirect detection. Indirect UV detection was employed in this study to visualize anions which have little and no chromophore. Through indirect detection, anions were recorded as positive peak while cations were recorded as negative peak. For reversing the direction of electroosmotic flow (EOF) from cathode to anode, a cationic surfactant that comprised of cetyltrimethylammonium hydroxide (CTAH) was added to the BGE.

A simultaneous separation of both anions ( $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{SCN}^-$ ) and cations ( $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ ) were successfully achieved using 2,6-PDC. Four anions and three cations were eluted within 7 minutes (Figure 1). Although the cations were detected as negative peaks, the PDC electrolyte provided a satisfactory separation of all seven components. All peaks were baseline separated except for  $\text{Cl}^-$  and  $\text{NO}_3^-$  peaks that were not well resolved. Another peak that eluted between  $\text{Fe}^{2+}$  and  $\text{Ca}^{2+}$  peaks indicated the presence of a solvent peak by comparison with the electropherogram of the blank extract.

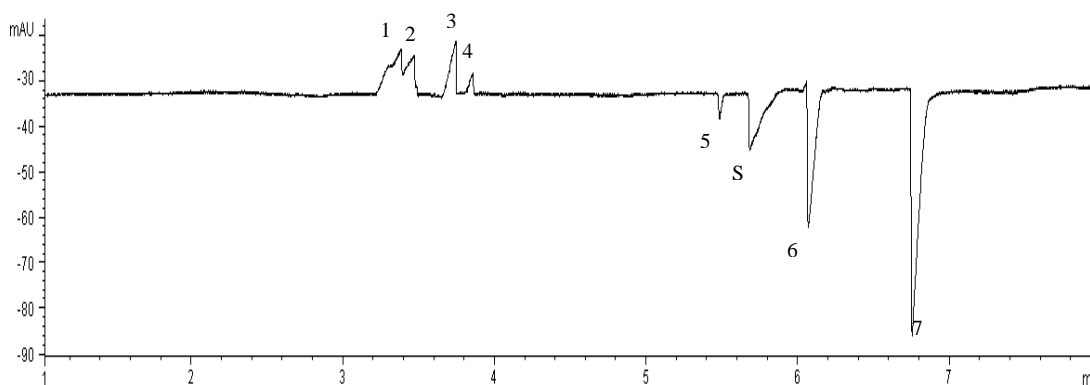


Figure 1: Electropherogram of inorganic anions and cations. Concentration of analytes at 100 ppm. Conditions: fused silica capillary (52 cm x 50  $\mu\text{m}$  I.D.); potential: -20kV; capillary temperature: 15°C; electrolyte: 25 mM 2,6-PDC and 0.5 mM CTAH (in 25% methanol); buffer pH 4.7; injection: 6.0 s x 50.0 mbar; wavelength: 350 nm, 20 ref. 230 nm, 10 ref. Peak identification: (1)  $\text{Cl}^-$ ; (2)  $\text{NO}_3^-$ ; (3)  $\text{SO}_4^{2-}$ ; (4)  $\text{SCN}^-$ ; (5)  $\text{Fe}^{2+}$ ; (6)  $\text{Ca}^{2+}$ ; (7)  $\text{Fe}^{3+}$ , (S) solvent.

### Optimization of CE Separation

#### i. Effect of BGE pH

Buffer pH would significantly have an effect on EOF because of an increase in the dissociation of silanol and silanoate group on the inner capillary wall [16]. As shown in Figure 2, migration time for most of the analytes generally increased with an increase in pH. However, a change of separation selectivity was obtained for  $\text{Ca}^{2+}$  and  $\text{Fe}^{3+}$  ions at pH 3.7 and pH 4.7.  $\text{Fe}^{3+}$  migrated earlier than  $\text{Ca}^{2+}$  at pH 3.7, but migrated later than  $\text{Ca}^{2+}$  at pH 4.7. This is because 2,6-PDC is an ionisable compound in which its ligand concentration depends on pH. Increasing pH will favor the protonated ligand concentration and also increases the degree of metal complexation. Besides that, pH changes will affect both separation selectivity and detection sensitivity [17].

An optimization of electrolyte pH was seen between pH 3.7 to 5.7 where the migration times of analytes were more stable. From Figure 3, it can be seen that  $\text{Ca}^{2+}$  could not be detected beyond pH 7.7 while,  $\text{Fe}^{2+}$  could not be detected at pH lower than 3.7. Therefore, pH 4.7 was selected as the optimized pH for subsequent experiment.

#### ii. Effect of PDC Concentration

Concentration of buffer also has an influence on EOF without altering the selectivity as pH. By increasing the buffer concentration, it will lower the EOF while lower buffer concentration will give shorter analysis time [18]. With a constant concentration of 0.5 mM CTAH in the electrolyte run, the concentration of 2,6-PDC was varied from 10 to 30 mM at pH 4.7 (Figure 3). Migration time of anions increased slightly with increasing BGE concentration while migration time of cations were found to increase significantly with increasing buffer concentration.

At a BGE concentration of 10 mM, the  $\text{NO}_3^-$  peak was difficult to observe and was not separated completely from  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  peaks while  $\text{Fe}^{2+}$  peak appeared closely to the solvent peak (Figure 3A). In addition, at 30 mM BGE concentration, the  $\text{SO}_4^{2-}$  and  $\text{SCN}^-$  peaks were not resolved and an increase in baseline noise was observed (Figure 3E). Under all BGE concentration employed,  $\text{Cl}^-$  and  $\text{NO}_3^-$  peaks were not separated completely. This could be due to insufficient length of the capillary used. However, resolved anionic peaks of  $\text{Cl}^-$  and  $\text{NO}_3^-$  were observed at 25 mM of 2,6-PDC. Hence 25 mM buffer concentration at pH 4.7 was selected as the optimum BGE condition.

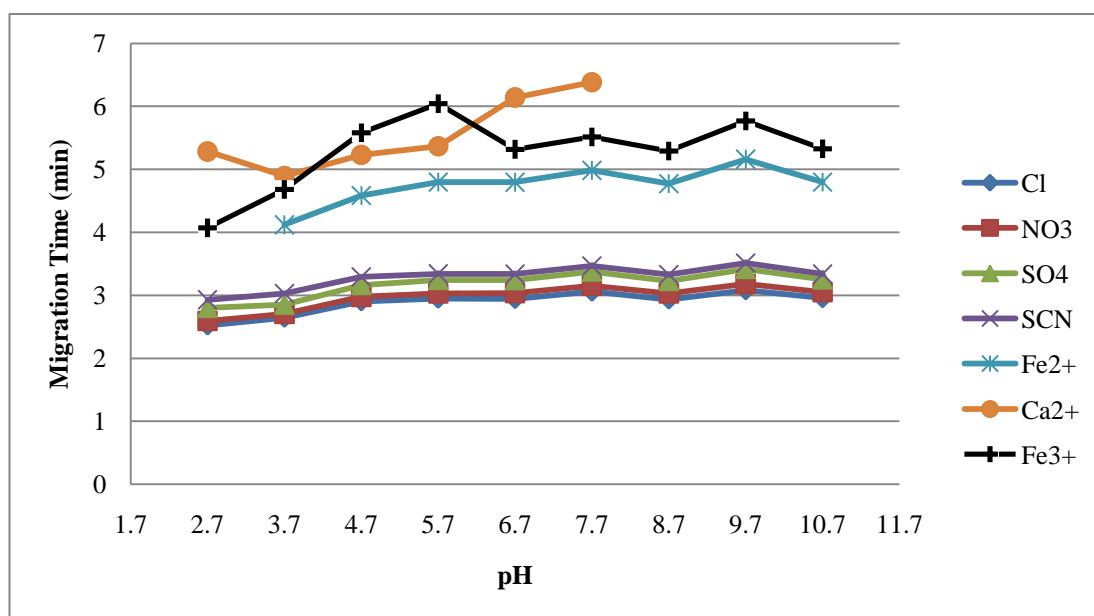


Figure 2: Effect of electrolyte pH on migration time

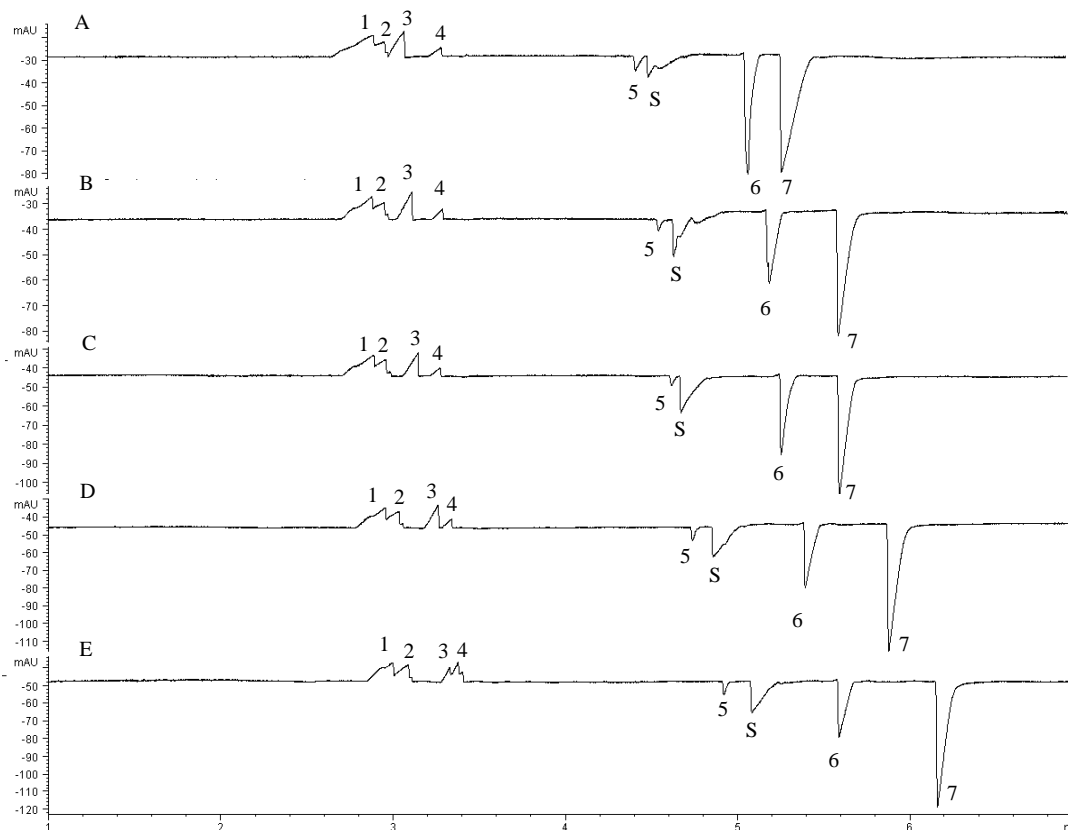


Figure 3: Effect of electrolyte concentration on peak separation. Concentrations of analytes at 100 ppm. Potential: -20kV; capillary temperature: 20°C; electrolyte: 0.5 mM CTAH with 2,6-PDC. (A) 10 mM, (B) 15 mM, (C) 20 mM, (D) 25 mM, and (E) 30 mM, pH 4.7. Peak identification: (1) Cl<sup>-</sup>; (2) NO<sub>3</sub><sup>-</sup>; (3) SO<sub>4</sub><sup>2-</sup>; (4) SCN<sup>-</sup>; (5) Fe<sup>2+</sup>; (6) Ca<sup>2+</sup>; (7) Fe<sup>3+</sup>, (S) solvent.

iii. Effect of Potential

The effect of potential was studied by the varying voltage from 15 kV to 25 kV as shown in Figure 4. Results indicated that by increasing the applied potential, the analysis time was typically reduced. In

contrast, at lower negative potential, the migration time was increased. In this study, although separation with short migration time was observed at -25 kV, adequate separation was observed at -20 kV with less current produced. Therefore, a potential of -20 kV was selected as the optimal running potential.

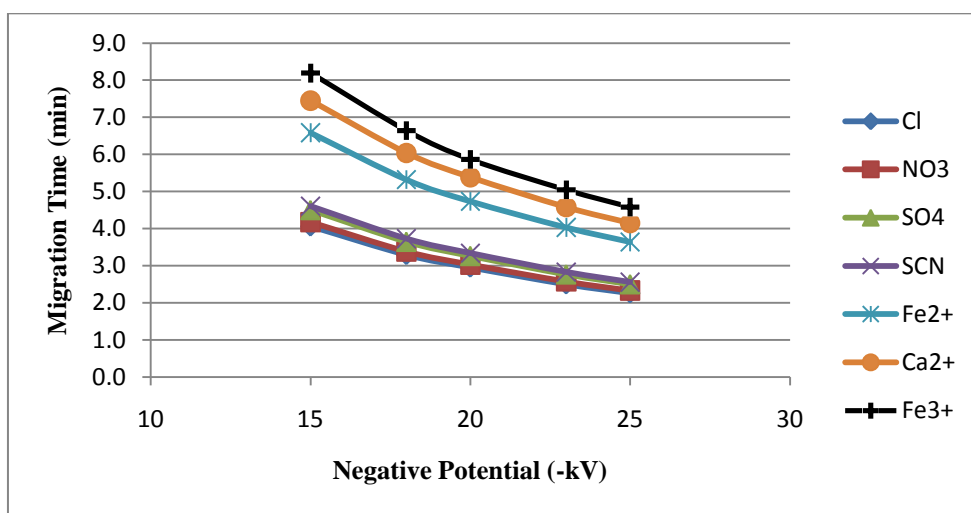


Figure 4: Effect of applied potential on migration time.

iv. Effect of Temperature

Figure 5 illustrates the migration times of target analytes in the temperature range from 15 to 30°C. Migration time was found to decrease with increasing temperature. A noisier baseline was also observed at higher temperature. A rise in temperature causes an increase in EOF due to a decrease in the

viscosity of the buffer. An increase of 1°C will generally reduce the viscosity of water by 2.4% [16]. Therefore, temperature was also considered as important in separation since both analyte mobility and level of EOF are temperature related. In this study, baseline drifts were observed at temperatures approaching 30°C. An optimum applied temperature for the best separation was therefore selected at 15°C.

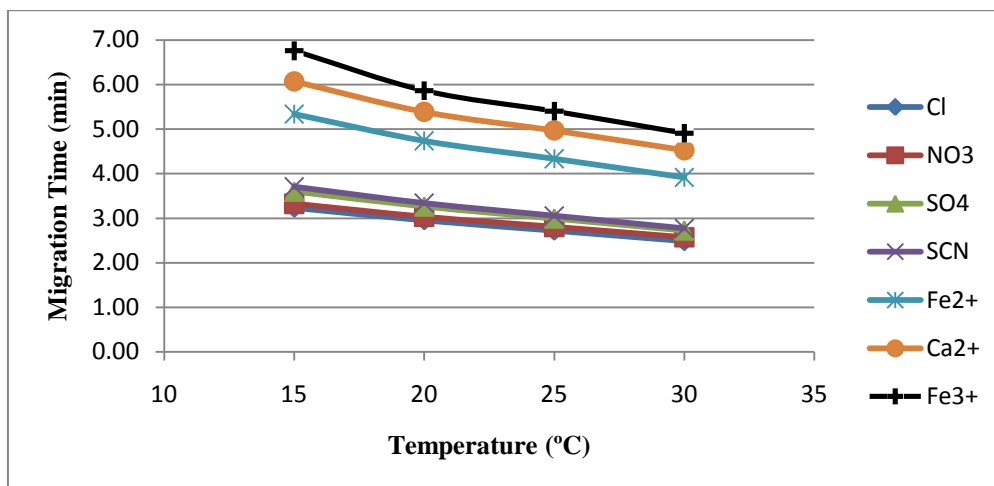


Figure 5: Effect of temperature on migration time.

Method Validation and Repeatability

A calibration plot of anions and cations was generated to determine the concentration of analyte ions in post-blast residues. Calibration curves were plotted for the respective inorganic ions within the concentration range of 20 to 100 ppm. The correlation coefficients ( $r^2$ ) and detection limits (LODs) are listed in Table 1. The correlation coefficients ( $r^2$ ) of each calibration curve were excellent ( $> 0.986$ ) that proved good linearity of the

present method. The detection limits for anions were in the range from 2 ppm to 15 ppm, while cations were in the range from 0.4 ppm to 3 ppm using a 50 mbar/s pressure injection at a signal to noise ratio of 2:1. Limits of detection from this study were compared with previous studies and it found that LODs in this study were much lower except for  $Ca^{2+}$ . The differences of LODs obtained from the present study and the earlier studies (Table 1) may be due to the different parameters employed in CE method.

Table 1: Correlation coefficients and detection limits of target analytes

Ion	Correlation coefficient ( $r^2$ )	LOD (ppm)	
		This work	Other work#
Cl-	0.990	2.0	8.0a
NO3-	0.996	3.0	11.0a
SO4 <sup>2-</sup>	0.988	3.0	9.0a
SCN-	0.986	15.0	-
Ca <sup>2+</sup>	0.995	2.0	0.4b
Fe <sup>2+</sup>	0.987	0.4	0.6c
Fe <sup>3+</sup>	0.988	3.0	6.0d

#References: <sup>a</sup>Jimidar *et al.* [20]  
<sup>b</sup>Chen and Naidu [17]  
<sup>c</sup>Lutzenkirchen and Lovgren [21]  
<sup>d</sup>Harrold *et al.* [22]

A method is considered precise when reproducible migration times and peak areas are obtained. Precision can be determined by performing repetitive analyses of the same sample within a system under the same condition and calculating the relative standard deviation. A standard ion mixture of 100 ppm was injected three times per day and also in six consecutive days respectively. The reproducibility of the method is tabulated in Table 2. The reproducibility of migration time was found to be better than 0.18% for within day variation. For day-to-day variation, the reproducibility of migration time was found to be better than 0.31%. In addition, the reproducibility of peak areas was found to be

better than 2.97% for within-day and 3.60% for day-to-day variation (Table 2). The results show good reproducibilities obtained for both migration time and peak area as reflected by the % RSD for within day (n=3) and day-to-day (n=6).

Determination of other cations including alkaline metal was also studied; however, no peaks were detected. This is because there is no change in UV absorbance between cation-complexes and PDC electrolyte. These cations might form negative complexes with PDC but due to its lower charge, the negative complexes could not be observed [13, 15].

Table 2: Within day and day-to-day reproducibility of target analytes

Ion	% RSD Migration Time		% RSD Peak Area	
	Within day	Day-to-day	Within day	Day-to-day
Cl <sup>-</sup>	0.031	0.236	2.206	1.431
NO <sub>3</sub> <sup>-</sup>	0.064	0.181	2.103	2.677
SO <sub>4</sub> <sup>2-</sup>	0.074	0.283	1.967	2.146
SCN <sup>-</sup>	0.042	0.312	2.973	0.437
Ca <sup>2+</sup>	0.085	0.208	0.826	2.439
Fe <sup>2+</sup>	0.049	0.222	1.238	0.548
Fe <sup>3+</sup>	0.181	0.181	2.623	3.598

#### Separation of ANFO Control Sample

In CE separation of ANFO control sample, only NO<sub>3</sub><sup>-</sup> was successfully identified (Figure 6). Ammonium ion could not be detected by CE due to its lower charge that causes the negative complexes not to

show any changes in UV absorbance [15]. Nitrate was present in large amount in the explosive mixture as indicated by the intense peak in the electropherogram. This peak indicated nitrate as an important component in ANFO explosive.

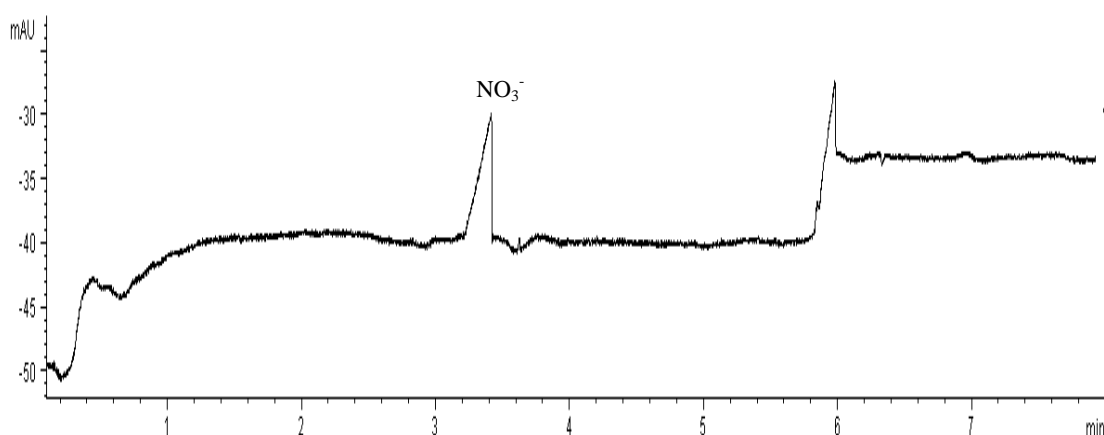
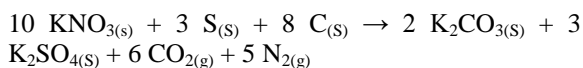


Figure 6: CE separation of ANFO control sample. CE conditions as in Figure 1.

*Post-Blast Residue Analysis*

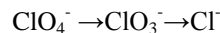
The developed method was applied to the determination of anions and cations in post-blast residue of black powder and ANFO. Black powder residue from three post-blast samples labeled BP-1, BP-2, and BP-3 were analyzed. CE separation revealed four ( $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{Ca}^{2+}$ ) ions in sample BP-1 and three ions ( $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{Ca}^{2+}$ ) in sample BP-2 and BP-3 (Figure 7).

Black powder is known to consist of potassium nitrate ( $\text{KNO}_3$ ), charcoal (C) and sulfur (S). Therefore the presence of nitrate ion ( $\text{NO}_3^-$ ) and sulfate ion ( $\text{SO}_4^{2-}$ ) are indicative of black powder as the explosive material used as follows.



The presence of calcium ( $\text{Ca}^{2+}$ ) and chloride ( $\text{Cl}^-$ ) can be explained through the principal ingredients of homemade explosives which consist of fuels and oxidizers. Fuels that are commonly used include sugar, sulfur, or metal such as calcium, aluminium, magnesium and zinc. These substances are mixed

with inorganic oxidizer such as salt containing nitrate, chlorate or perchlorate. During explosion, these chemicals undergo chemical reaction to produce ionic species in post-blast residue [2]. According to McCord *et al.* [6], the presence of chloride is a result of the chemical reduction of chlorate from perchlorate oxidizer as follows.



Analysis of ANFO residue deposited on cloth (cotton and nylon) was also conducted using the developed method. A minor peak due to nitrate ( $\text{NO}_3^-$ ) was identified in the cotton extract. For further confirmation of the small peak in cotton extract,  $\text{NO}_3^-$  standard of known concentration was spiked into cotton extract and CE analysis was carried out. The analysis showed enhancement in peak area that indicated the presence of  $\text{NO}_3^-$  in the cotton extract. Nitrate was not detected in nylon extract possibly due to low amounts of ANFO residue that adhered to nylon. Nylon is a man-made fiber which is smooth and renders the residue difficult to adhere onto the fiber. The presence of  $\text{NO}_3^-$  indicated the use of ANFO since the major ingredient in ANFO is ammonium nitrate.

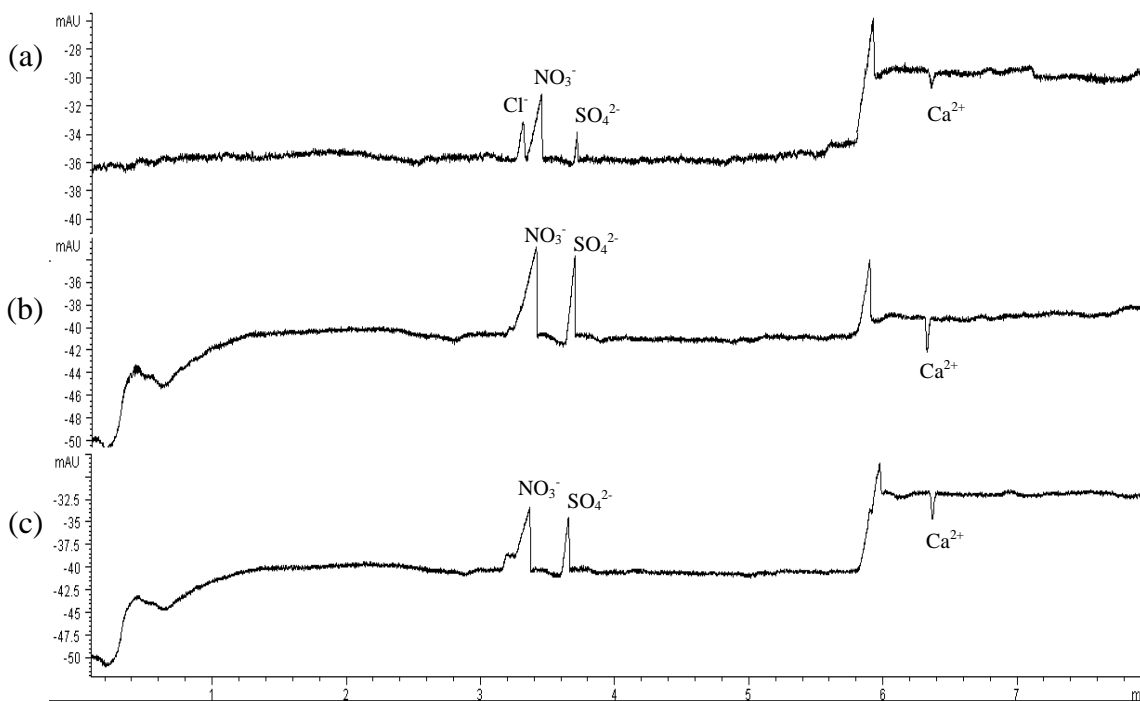


Figure 7: CE profile of black powder residue from three post-blast samples (a) BP-1, (b) BP-2, (c) BP-3. CE conditions as in Figure 1.



## Conclusions

A reliable and rapid method for simultaneous determination of cations and anions using CE has been developed. This method offers the advantages of fast analysis, minimal sample consumption and capable of separating inorganic anions and cations simultaneously in a single run and with good linearity and reproducibility. The developed CE technique was applied for the detection of selected inorganic ions in several post-blast residues of explosive samples. Although CE could not detect several cations of forensic interest ( $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{NH}_4^+$ ), detection of three anions ( $\text{Cl}^-$ ,  $\text{NO}_3^-$  and  $\text{SO}_4^{2-}$ ) along with three cations ( $\text{Fe}^{2+}$ ,  $\text{Ca}^{2+}$  and  $\text{Fe}^{3+}$ ) indicated the use of black powder in the explosive. Meanwhile, detection of  $\text{NO}_3^-$  indicated the use of ANFO. However, the detection limits obtained (0.4-15 ppm) does not yet satisfy the requirements for real post-blast residue analysis. CE pre-concentration of analytes based on stacking or other techniques can be further developed to address this problem.

## Acknowledgement

The authors would like to thank Royal Malaysia Police for assistance in the explosive sampling; Universiti Pertahanan Nasional Malaysia for financial support to Nor Laili-Azua; Ministry of Higher Education Malaysia for financial research aid; and Faculty of Science, Universiti Teknologi Malaysia for research facilities.

## References

1. Johns, C., Shellie, R. A., Potter, O. G., O'Reilly, J. W., Hutchinson, J. P., Guijt, R. M., Breadmore, M. C., Hilder, E. F., Dicinoski, G. W., and Haddad, P. R. (2008). Identification of Homemade Inorganic Explosives by Ion Chromatographic Analysis of Post-Blast Residue. *J. Chromatogr. A*, 1182: 205-214.
2. Hutchinson, J. P., Evenhuis, C. J., Johns, C., Kazarian, A. A., Breadmore, M. C., Macka, M., Hilder, E. F., Guijt, R. M., Dicinoski, G. W. and Haddad, P. R. (2007). Identification of Inorganic Improvised Explosive Devices by Analysis of Postblast Residue Using Portable Capillary Electrophoresis Instrumentation and Indirect Photometric Detection with a Light-Emitting Diode. *Anal. Chem.* 79: 7005-7013.
3. Hargadon, K. A. and McCord, B. R. (1992). Explosive Residue Analysis by Capillary Electrophoresis and Ion Chromatography. *J. Chromatogr.* 602: 241-247.
4. Doyle, J. M. and McCord, B. R. (1998). Novel Electrolyte for the Analysis of Cations in Low Explosive Residue by Capillary Electrophoresis. *J. Chromatogr. B.* 714: 105-111.
5. Hopper, K. G., LeClair, H. and McCord, B. R. (2005). A Novel Method for Analysis of Explosives residue by Simultaneous Detection of Anions and Cations via Capillary Electrophoresis. *Talanta.* 67: 304-312.
6. McCord, B. R., Hargadon, K. A., Hall, K. E. and Burmeister, S. G. (1994). Forensic Analysis of Explosives using Ion Chromatographic Methods. *Anal. Chim. Acta.* 288: 43-56.
7. Olofson, S. (2009). *Identification of Peroxide Explosives and Traditional Explosive Anion by Capillary Electrophoresis.* Oklahoma State University: Master Thesis.
8. Ahmad U.K., Ong S.T., Ghazali M. F., Yew C. H., and Abdullah M. K. (2011). Analysis of Anionic Post-Blast Residues of Low Explosives from Soil Samples of Forensic Interest, *Malays. J. Anal. Sci.*, 15, (2), 213-226.
9. Cruces-Blanco, C., Gamiz-Gracia, L. and Garcia-Campana, A. M. (2007). Application of Capillary Electrophoresis in Forensic Analytical Chemistry. *Trends Anal. Chem.* 26 (6): 215-226.
10. Skoog, D. A., Holler, F. J. and Nieman, T. A. (1998). *Principle of Instrumental Analysis.* 5<sup>th</sup> edition. U.S.: Brook/Cole Thomson Learning.
11. Padaruskas, A., Olsauskaite, V. and Paliulionyte, V. (1998). Simultaneous Determination of Inorganic Anions and Cations in Waters by Capillary Electrophoresis. *J. Chromatogr. A*, 829: 359-365.
12. Tsuda, T. (1987). Modification Of Electroosmotic Flow With Cetyltrimethylammonium Bromide In Capillary Zone Electrophoresis. *J. High Resolut. Chromatogr.*, 10: 622-624.
13. Soga, T. and Ross, G. A. (1999). Simultaneous Determination of Inorganic Anions, Organic Acids and Metal Cations by Capillary Electrophoresis. *J. Chromatogr. A*, 834: 65-71.
14. Chen, Z., Tang, C. and Xu, J. (1999). Non-Suppressed Conductivity And Indirect UV Detection Of Carboxylic Acids In Environmental Samples By Ion-Exclusion Chromatography Using 2,6-Pyridinedicarboxylic Acidic Eluent. *J. Chromatogr. A*, 859: 173-181.
15. Chen, Z.L., Naidu, R., and Subramaniam, A. (2001). Separation Of Chromium (III) And Chromium (VI) By Capillary Electrophoresis Using 2,6-Pyridinedicarboxylic Acid As Pre-Column Complexation Agent. *J. Chromatogr. A*, 927:219-227.

16. Baker, D. R. (1995). *Capillary Electrophoresis: Technique in Analytical Chemistry*. Canada: John Wiley & Sons, Inc.
17. Chen, Z.L. and Naidu, R. (2002). On-Column Complexation of Metal Ions Using 2,6-Pyridinecarboxylic Acid And Separation Of Their Anionic Complexes By Capillary Electrophoresis With Direct UV Detection. *J. Chromatogr. A*, 966: 245-251.
18. Chen, Z. L. and Adams, M. A. (1999). 2,6-Pyridinedicarboxylic Acid As An Eluent For UV And Conductivity Detection Of Inorganic Anions, Magnesium And Calcium In Water By Ion Chromatography. *Chromatographia*, 49 (9): 496-502.
19. Khairiah, H. (2010). *Detection of Anionic Components from Fragments of Home-Made Explosive Residues*. Universiti Teknologi Malaysia: MSc. Dissertation.
20. Jimidar, M., Hamoir, T.P., Foriers, A., and Massart, D.L. (1993). Comparison of Capillary Electrophoresis Zone Electrophoresis With High-Performance Liquid Chromatography For The Determination Of Additives In Foodstuff. *J. Chromatogr.* 636: 179-186.
21. Lützenkirchen, J. and Lövgren L. (1998): Experimental Study of Arsenite Adsorption to Goethite. *Mineral Sciences*, 62A, 927-928.
22. Harrold, M.P., Wajtusik, M.J., Riviello, J. and Henson, P. (1993). Parameters Influencing Separation and Detection of Anions by Capillary Electrophoresis. *J. Chromatogr.* 640: 463-471.

*Additional information and reprint requests:*

Umi Kalthom bt Ahmad  
(Email: [umi@kimia.fs.utm.my](mailto:umi@kimia.fs.utm.my))  
Department of Chemistry  
Faculty of Science  
Universiti Teknologi Malaysia  
81310 UTM Skudai, Johor, Malaysia