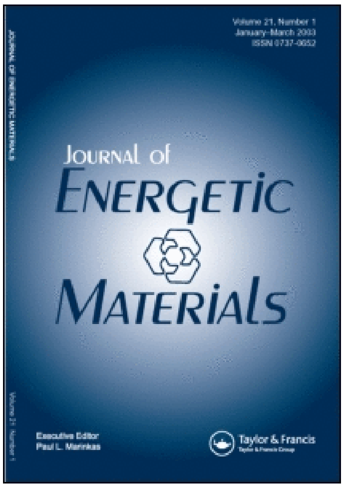
This article was downloaded by:
On: *16 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Energetic Materials

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713770432

Analysis of cobalt based explosives by capillary electrophoresis

Stuart A. Oehrle[a]; Tom Massis[b]
[a] Waters Corporation, Milford, MA [b] Sandia National Laboratories, Albuquerque, NM

To cite this Article Oehrle, Stuart A. and Massis, Tom(1997) 'Analysis of cobalt based explosives by capillary electrophoresis', Journal of Energetic Materials, 15: 2, 125 — 137

To link to this Article: DOI: 10.1080/07370659708216077

URL: http://dx.doi.org/10.1080/07370659708216077

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

ANALYSIS OF COBALT BASED EXPLOSIVES BY CAPILLARY ELECTROPHORESIS*

Stuart A. Oehrle*
Waters Corporation
34 Maple St.
Milford, MA 01757

Tom Massis
Sandia National Laboratories
Albuquerque, NM 87185

ABSTRACT

Cobalt based explosives have received much attention for possible uses in DOE weapons system. One such compound, tetraaminebis(5-nitro-2H-tetrazolato-N^2) cobalt (III) perchlorate, (BNCP) is being investigated. Analysis of BNCP as well as it's precursors is of importance in assessing the amount of impurities which may be present and potentially interfere in the overall performance of the explosive. Capillary electrophoresis (CE) offers a means of rapidly analyzing for these components. Capillary electrophoresis (CE) was used to investigate ionic impurities in BNCP, as well as the precursors used to synthesize the BNCP explosive. Analysis of cationic contaminants in several precursors, CTCN and CPCN, as well as in BNCP could be done using indirect UV detection at 185nm in less than 10 minutes by CE.

***Presented at the 5th International Symposium on the Analysis and Detection of Explosives, Washington, D.C., December 1995**

Journal of Energetic Materials Vol. 15, 125-137 (1997)
Published in 1997 by Dowden, Brodman & Devine, Inc.

INTRODUCTION

Since the early 1970's, it has been known that certain 5-substituted pentaaminetetrazolatocobalt(III) perchlorates would undergo deflagration-to-detonation transition (DDT) under appropriate confinement. Within this series the 5-cyanotetrazolato complex, known as CP, has been used in DOE weapons since 1979. More recently CP detonators have been used in oil field completion work as well as other DOE and DOD applications.(1)

In 1986, Bates reported a related compound, tetraaminebis(5-nitro-2H-tetrazolato-N^2)cobalt(III) perchlorate, as a possible replacement for lead azide and other priming materials.(2) This compound, referred to as BNCP, was found to undergo DDT in a smaller and less confining configuration than CP. BNCP also exhibited a higher explosive yield than CP as indicated by witness block testing and later by the VISAR method. Figure 1 shows the structures of CP and BNCP.

The synthesis of BNCP is accomplished with a 55-70% yield for the final BNCP product. However the greater challenge to the synthesis is the preparation of the precursor tetraaminecarbonatocobalt(III) nitrate (CTCN). Detailed examination of the product obtained found the presence of pentaaminecarbonatocobalt(III) nitrate (CPCN). Figure 2 shows the structure for the CTCN and CPCN compounds. Quality of the precursor CTCN for the synthesis of BNCP is critical in allowing for the highest yield of BNCP as well as the least amount of contamination. For this reason analytical methods for analyzing for CTCN, CPCN, and BNCP were investigated.

Capillary electrophoresis (CE) was chosen for the analysis since it is a separation technique which can easily separate compounds based on their overall charge. CE has been successfully used in the past for the analysis of small inorganic and organic ions.(3-6) Analysis of different lots of BNCP, CPCN and

CTCN samples was done with total analysis times of less than 10 minutes possible. Further, small cations (i.e. sodium, potassium, etc.) were also separated using this technique.

CHEMICALS and SAMPLE INFORMATION

The electrolyte used consisted of UV Cat-1, HIBA (hydroxyisobutyric acid), and crown ether. (Waters Corp., Milford, MA). Cation standards were prepared as concentrates from their salts (ACS grade or better). Plastic volumetric flask were used for all sample and standard preparation. High purity water was used for all preparation and dilution's (Millipore Corp., Bedford, MA). Various grades of BNCP, CTCN, and CPCN samples from different suppliers to Sandia were obtained and analyzed.

EXPERIMENTAL

Instrumentation:

The capillary electrophoresis (CE) system employed was the Quanta™ 4000E Capillary Ion Analyzer (Waters Chromatography, Milford, MA, U.S.A.). A Hg lamp was used for indirect UV detection at 185nm for cation analysis. AccuSep™ polyimide fused silica capillaries of dimension 75 m I.D. X 60cm were used throughout. Data acquisition was carried out with a Waters Millennium™ 2010 Chromatography Manager with a SAT/IN module connecting the CE to the data station with the signal polarity inverted from the CE.

The analysis was done using a hydrostatic injection for 12 seconds and an applied voltage of 20 kV.

Preparation of electrolytes:

High purity water (Milli-Q™) was used to prepare all solutions (Millipore, Bedford, MA, U.S.A.). The working electrolyte for cation analysis was a solution of 5.0 mM UV Cat-1, 6.5 mM HIBA (both from Waters) and 2.0 mM

18-Crown-6 (Aldrich Chemical, Milwaukee, WI, U.S.A.). All working electrolytes were prepared fresh daily and degassed prior to use.

RESULTS

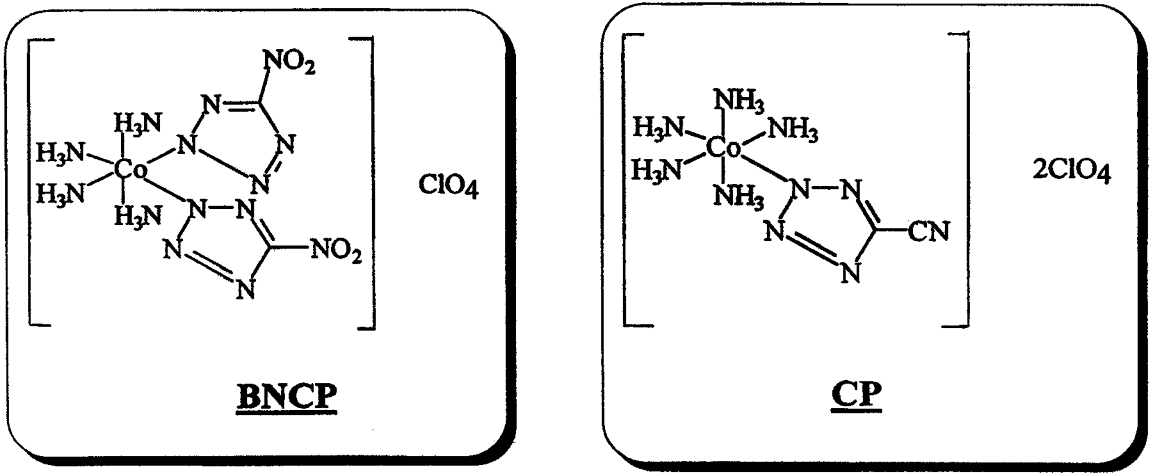
Figure 3 is an electropherogram of a cation standard showing the separation for alkali and alkaline earth cations and cobalt. Figure 4 is an electropherogram of a lot of CTCN showing the presence not only CTCN and other small cations but a large unidentified peak before ammonium. This unknown peak was not found in a highly purified sample of CTCN (figure 5). Both samples are at approximately the same concentration (based on mass of dry material weighed out). This unknown peak was found to be present in CPCN (figure 6) suggesting that CPCN is present in the less clean lot of CTCN (figure 4). This unknown peak is not found in the final BNCP products that were analyzed but in a crude (unclean) lot of BNCP several unknown peaks around the large sodium peak (figure 7) were found. These peaks may be small amines that from the synthesis itself or contaminants in the reaction flask itself that were not cleaned out. A purified lot of BNCP (figure 8) shows only BNCP and some minor contaminants present.

CONCLUSIONS

Capillary electrophoresis (CE) offers a means of rapidly evaluating the cleanliness and purity of various samples of the cobalt based explosive BNCP and it's precursors. Qualitative data on the presence of contaminants could be done using CE in less than 10 minutes.

FURTHER WORK

Further work on identifying the unknown contaminants and how they affect the overall performance of the explosive material will be done. The CE will continue to be used for evaluating the raw materials prior to synthesis.



BNCP

CP

Figure 1: Chemical structure of BNCP (tetraaminebis(5-nitro-2H-tetrazolato-N^2)cobalt(III) perchlorate) and CP (pentaamine(5-cyano-2H-tetrazolato-N^2)cobalt(III) perchlorate)

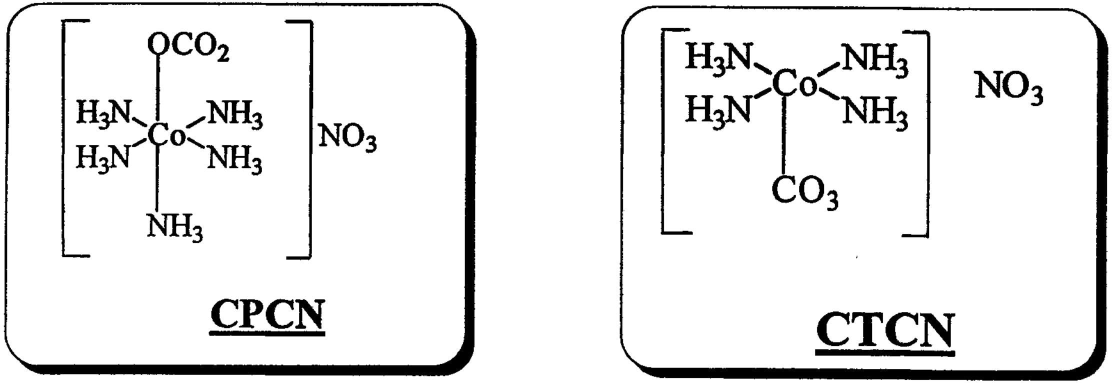


Figure 2: Chemical structure of CPCN (carbonatopentaaminecobalt(III) nitrate) and CTCN (carbonatotetraminecobalt(III) nitrate)

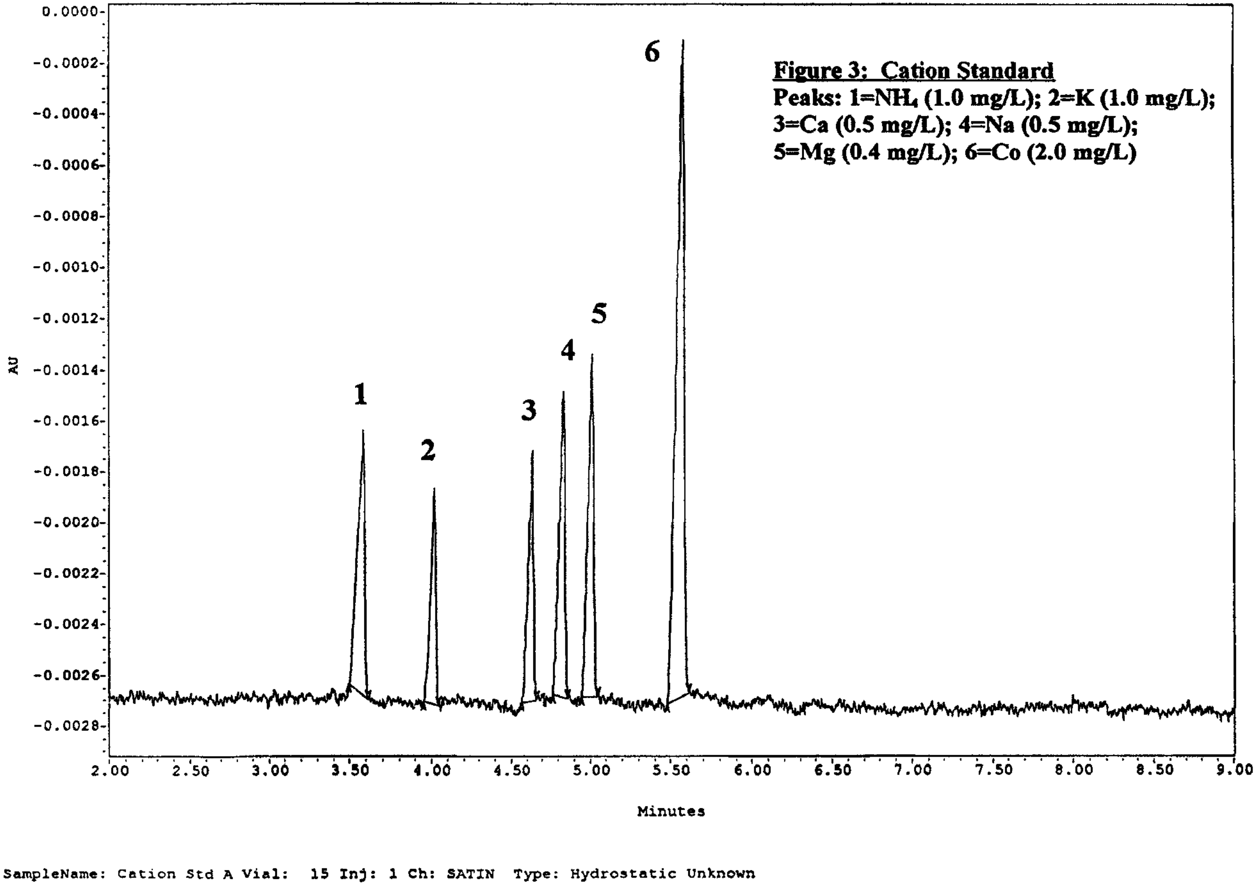


Figure 3: Cation Standard
Peaks: 1=NH_4 (1.0 mg/L); 2=K (1.0 mg/L); 3=Ca (0.5 mg/L); 4=Na (0.5 mg/L); 5=Mg (0.4 mg/L); 6=Co (2.0 mg/L)

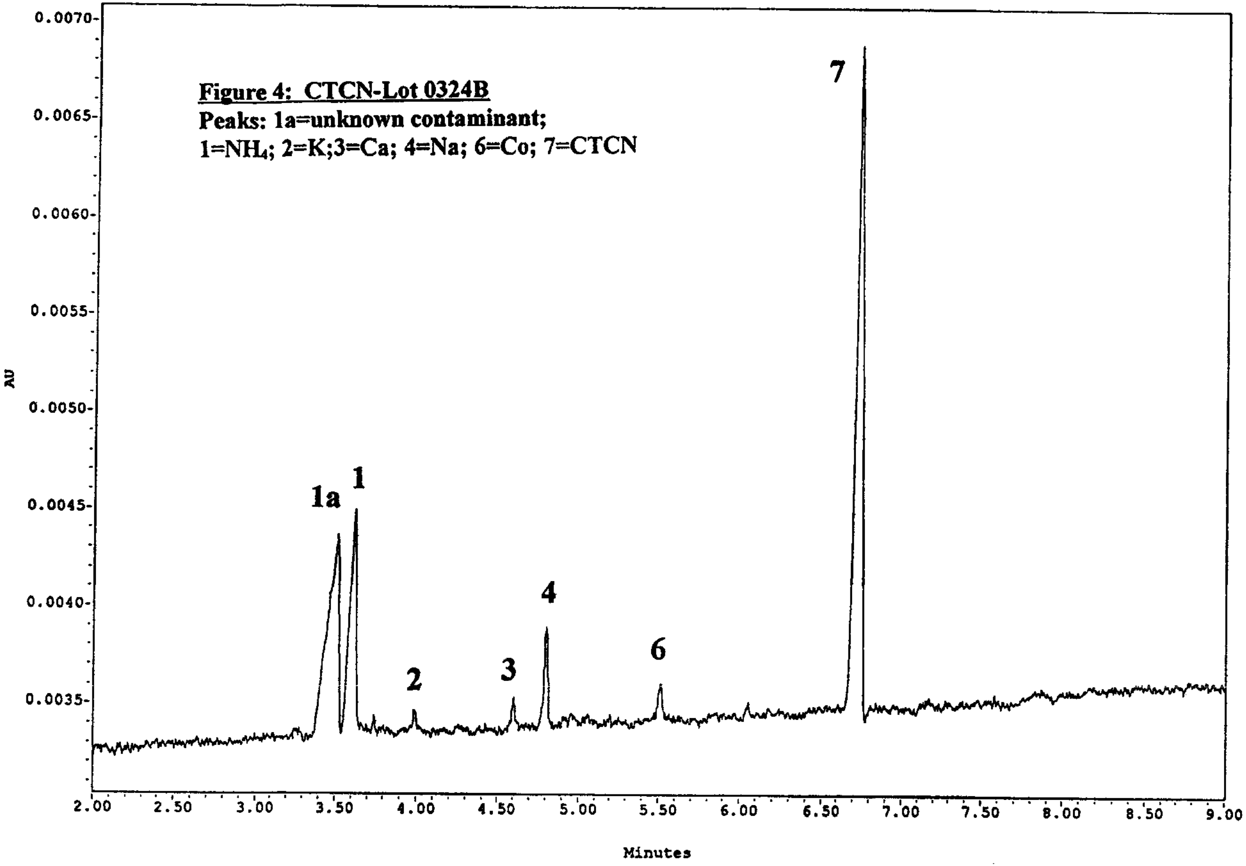


Figure 4: CTCN-Lot 0324B

Peaks: 1a=unknown contaminant;

1=NH_4; 2=K;3=Ca; 4=Na; 6=Co; 7=CTCN

SampleName: CTCN #6 (0.1-10mL) Vial: 4 Inj: 1 Ch: SATIN Type: Hydrostatic Unknown

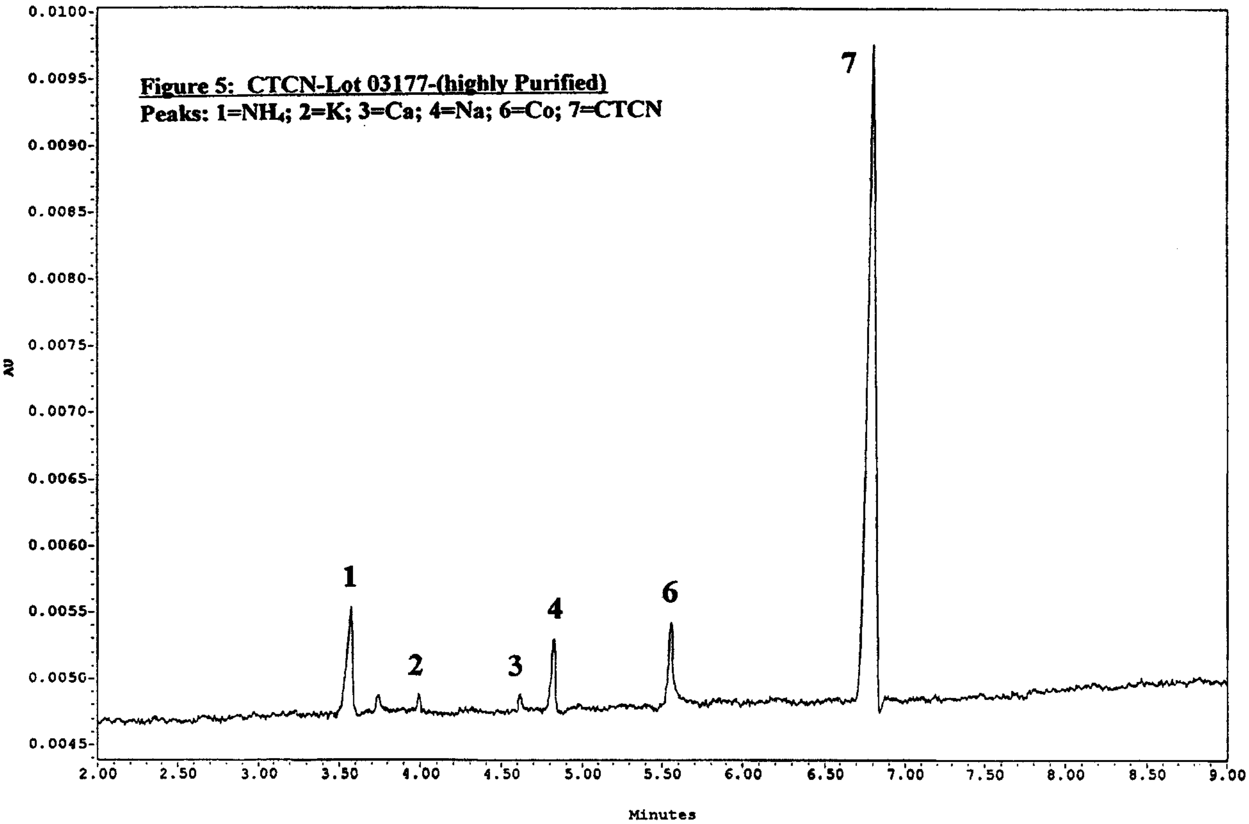


Figure 5: CTCN-Lot 03177-(highly Purified)
Peaks: 1=NH_4; 2=K; 3=Ca; 4=Na; 6=Co; 7=CTCN

SampleName: CTCN #8 (0.1-10mL) Vial: 6 Inj: 1 Ch: SATIN Type: Hydrostatic Unknown

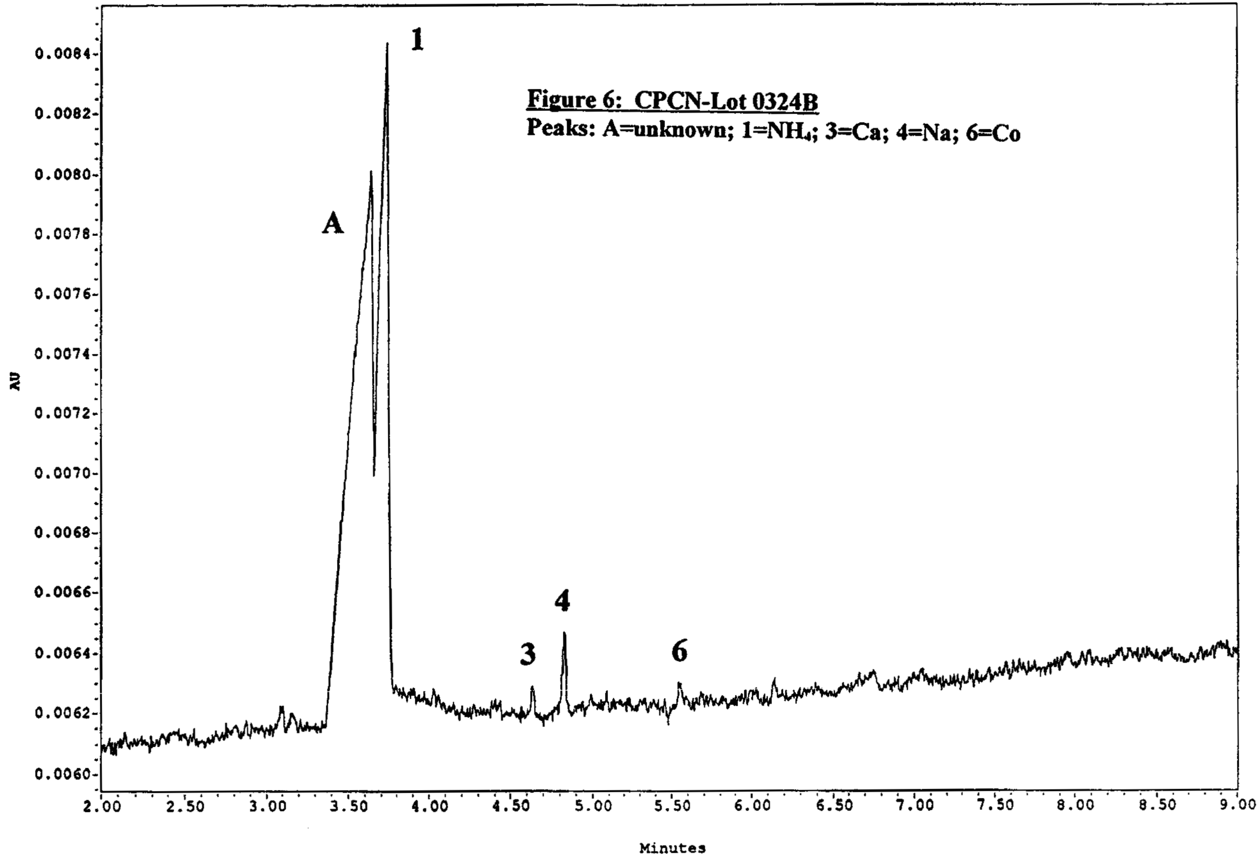


Figure 6: CPCN-Lot 0324B

Peaks: A=unknown; 1=NH_4; 3=Ca; 4=Na; 6=Co

SampleName: CPCN #7 (0.1-10mL) Vial: 8 Inj: 1 Ch: SATIN Type: Hydrostatic Unknown

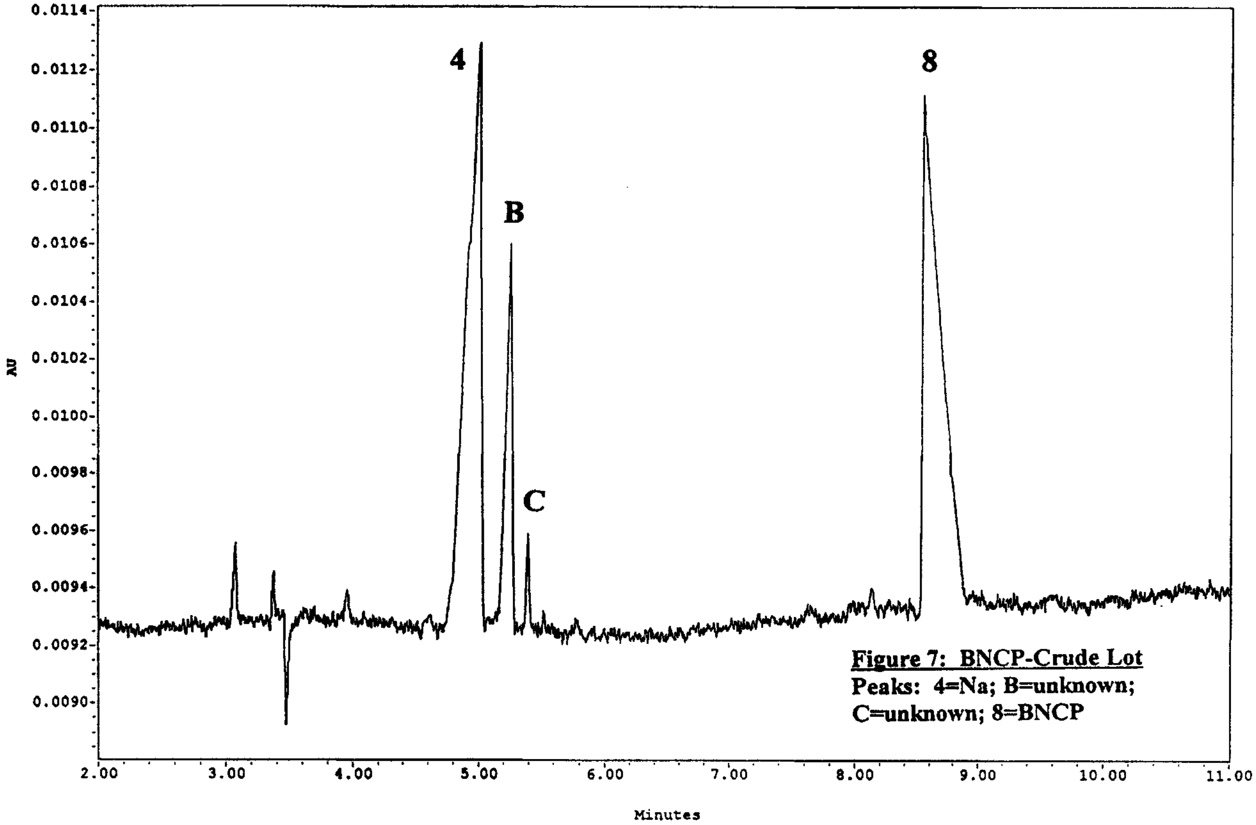


Figure 7: BNCP-Crude Lot
Peaks: 4=Na; B=unknown;
C=unknown; 8=BNCP

SampleName: BNCP #1 (0.05-0.5mL)-30 sec Vial: 12 Inj: 1 Ch: SATIN Type: Hydrostatic Unknown

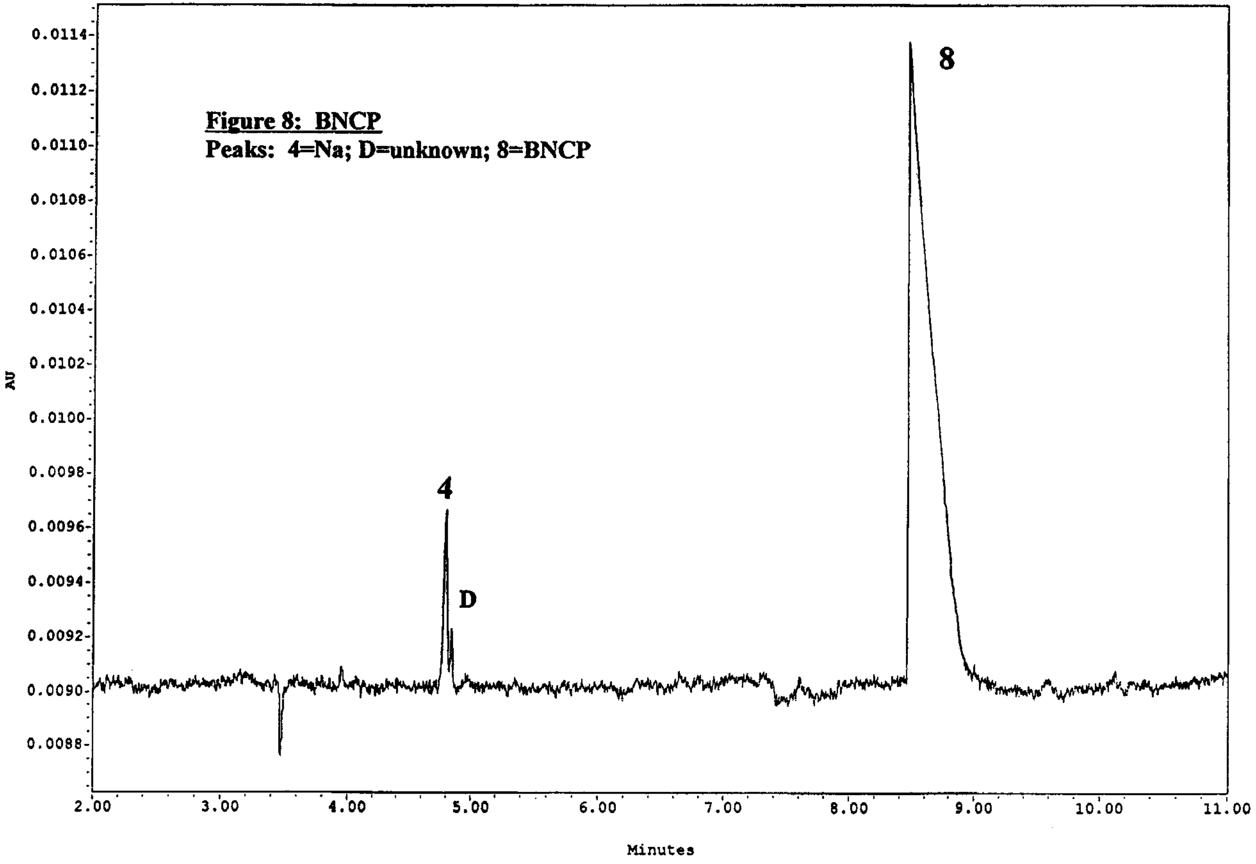


Figure 8: BNCP

Peaks: 4=Na; D=unknown; 8=BNCP

SampleName: BNCP #5 (0.05-0.5mL)-30 sec Vial: 11 Inj: 1 Ch: SATIN Type: Hydrostatic Unknown

REFERENCES

1. Fronabarger, J.; Schuman, A., Chapman, R.D.; Fleming, W.; Sanborn, W.B.; and Massis, T.; "Chemistry and development of BNCP, a novel DDT explosive"; Presented at ADPA, New Orleans, March, 1994.
2. Bates, L.R.; "The potential of tetrazoles in initiating explosive systems"; Proceeding's of the 13th symposium on explosives and pyrotechnics, Hilton Head, December, 1986.
3. Jandik, P., and Bonn, G.; "Capillary electrophoresis of small molecules and ions"; VCH Publishers, New York, (1993)
4. Oehrle, S.A. ; "Analysis of cationic ingredients and degradation products in liquid gun propellants by capillary ion electrophoresis"; *J. Energetic Materials*, vol. **12**, (1994), p. 197.
5. Weston, A.; Brown, P.R.; Jandik, P.; Jones, W.R.; and Heckenberg, A.L.; "Factors affecting the separation of inorganic metal cations by capillary electrophoresis"; *J. Chromatogr.*; **593**, (1992), p. 289.
6. Oehrle, S.A.; Blanchard, R.D.; Stumpf, C.L.; and Wulfeck, D.L.; "Environmental monitoring of wastewater using capillary ion electrophoresis"; J. Chromatogr.; **680**, (1994), p. 645.