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High-Pressure Liquid Chromatographic Method for CP Amide Analysis

Vincent M. Loyola, Jean Womelsduff

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HIGH-PRESSURE LIQUID CHROMATOGRAPHIC METHOD
FOR CP AMIDE ANALYSIS

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ABSTRACT

A method has been developed for the analysis of the 5-carboxamidotetrazolatopentaamminecobalt(III) perchlorate contaminant of the explosive CP (2-(5-cyanotetrazolato)-pentaamminecobalt(III) perchlorate). The analysis utilizes a Zn/Hg reduction of the cobalt(III) complexes to allow a high pressure liquid chromatographic (HPLC) analysis of the reaction products; the product analysis is directly related to the weight percent of impurity present in the explosive. The technique is described and shortcomings pointed out.

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INTRODUCTION

The explosive CP (2-(5-cyanotetrazolato)pentaamminecobalt(III) perchlorate), Figure 1a, is a high energy inorganic complex which has been developed by Sandia National Laboratories for use in detonating components. The CP is produced, under contract to Sandia, by Unidynamics/Phoenix, Inc. and subjected to qualifying tests according to established criteria.^{1,2} One of the criteria which has been established for product acceptance is the percentage of 5-carboxamidotetrazolatopentaamminecobalt(III) perchlorate (CP amide), Figure 1b, which will be accepted as an impurity in each lot of CP produced. CP amide is present in the CP as a contaminant which is produced during the synthesis via hydrolysis

FIGURE 1. a: 2-(5-cyanotetrazolato)pentaamminecobalt(III) perchlorate.

b: 5-carboxamidotetrazolatopentaamminecobalt(III) perchlorate. The exact coordination of the heterocyclic is not known but is suspected to be at the 2 position.

of the cyano group of CP. The CP amide thus produced is very difficult to remove completely from the CP, and typically a CP amide content of approximately 3 weight percent remains in the final product. Successive recrystallizations do not lower the CP amide content significantly and result in a considerably decreased yield of CP.³ Because of the difficulty and cost involved in obtaining extremely pure CP and because the CP amide, in the amounts at which it is typically present, has nowhere been shown to be detrimental to the performance properties of CP, the maximum limit of CP amide tolerable in CP has been set at 5 weight percent.² This criterion, therefore, necessitates a method by which CP amide can be quantitated in the CP.

Two methods are currently employed for measuring the quantity of CP amide present in CP, and only one is accepted by Sandia. The first method is a thin layer chromatographic (TLC) technique, employed at Unidynamics as a screening test,¹ which utilizes a sulfide spray as a cobalt visualizer to develop the chromatograms. Once developed, the chromatograms are visually evaluated, and the unknown CP amide content of the CP samples is estimated by comparison to other chromatograms of known concentrations of CP amide. This method gives surprisingly good estimates of CP amide content but does have some shortcomings. The most serious shortcoming is its approximate nature, but there are others such as tailing of the chromatograms which could also give rise to error in the estimate. For these reasons this method is only used as a screening test.

The second, and only currently accepted method, is an infrared (I.R.) technique.⁴ This technique relies on the complete deuteration of both the CP and CP amide complex in order to enhance

the sensitivity of the method. Deuteration of the complexes results in a shift of the NH_3 I.R. active bands from an interfering to a non-interfering position in the I.R. spectrum of CP-CP amide mixtures. With the interference removed, the carbonyl stretch of the carboxamide group can then be utilized for quantitative evaluation of the percentage of CP amide present. This technique gives good results when the procedure is carried out properly, but it has some shortcomings. The technique is sensitive to pH during deuteration, to the rate of solvent removal after complete deuteration, and to whether the solvent is removed by freeze drying or pumped off at above freezing temperatures. However, its most serious disadvantage is its time consuming nature since it takes from 24 to 30 hours to completely deuterate the complexes and to completely remove the solvent from the solids. Because of the difficulties with the method, the results obtained by different laboratories have at times been in disagreement.

It is, therefore, desirable to establish a method for CP amide analysis which is both more rapid and more likely to be reproducible at other laboratories. The present work explores the possibility of using HPLC (High Pressure Liquid Chromatography) as a method which will meet both criteria. HPLC separation of CP and CP amide has been attempted earlier.^{5,6} However, because of the molecular similarities of the two complexes, little success has been attained. The present report describes the successful application of a technique in which the complexes are first reduced to produce solutions of the free cyanotetrazole and carboxamido tetrazole, after which the ligands are separated by means of HPLC with good resolution.

EXPERIMENTAL

Reduction of Co(III) Complexes: The cobalt(III) complexes were reduced over Zn/Hg amalgam in 0.45 M HClO_4 in the following manner. The Co(III) complexes were made to a predetermined concentration in water ($\sim 5.0 \times 10^{-3}$ molar) and 25 ml of this solution was then transferred to a 50 ml erlenmeyer flask containing approximately 2 gm of Zn/Hg. The solutions were stirred with Teflon coated magnetic stirrers at a rate sufficient to give a good flow of solution through the amalgam, but slow enough to keep spattering of the solution to a minimum. One ml of concentrated HClO_4 (J. T. Baker Reagent grade, 11.7 molar) was added to the stirred solutions and the reduction allowed to proceed at room temperature for 40-60 minutes. The reactions were usually complete at the end of 40 minutes, but the reaction products did not appear to degrade with up to 60 minutes of reaction time. When the reaction was complete, the Zn/Hg was filtered out of the solution using Whatman Number 1 filter paper and the acidified solutions were injected onto the HPLC column.

Stirring efficiency is very important in the reduction of the Co(III) complex. Results obtained using the above procedure, but with inefficient stirring, were spurious and irreproducible. Considering that the reaction is a surface reaction and that the rate should be surface area and stirring rate dependent, one should not be surprised by the problems observed. The problem is avoided by taking care that the stirring is smooth and efficient, so that a good flow of solution through the amalgam is obtained.

Zn/Hg Reductant: The Zn/Hg reductant used in this study was prepared from 20 mesh granular zinc metal (Mallinkrodt Analytical Reagent) and mercuric chloride (J. T. Baker, Analyzed Reagent grade) in the following manner.

Approximately 80 grams Zn metal was placed in a medium porosity fritted glass Buchner funnel and rinsed with H_2O , which was pulled through by vacuum while the metal was stirred with a Teflon rod. The metal was then treated in a similar manner with 0.10 M HCl (J. T. Baker, Reagent grade) to remove oxides and was followed by rinsing with H_2O to remove HCl. The Zn/Hg was prepared by adding enough $HgCl_2$ solution (0.075 M $HgCl_2$ in 0.2 M $HClO_4$) to cover the cleaned metal. The metal was stirred for about 30 seconds and allowed to stand for 2-3 minutes before the $HgCl_2$ solution was pulled through the funnel by vacuum. The Zn/Hg was then rinsed with two 50 ml aliquots of H_2O and allowed to air dry on the funnel for several minutes. The Zn/Hg was then sandwiched between two pieces of clean filter paper and allowed to complete drying. The dry Zn/Hg was placed in a covered container and stored until used. Storage of the Zn/Hg for several days did not seem to affect its performance relative to freshly prepared Zn/Hg. The Zn/Hg was used only once and then discarded.

Chromatographic System: The instrument used for this work was a Varian Aerograph 4100 LC system modified to accept a Rheodyne Model 7125 syringe loading loop injector, and plumbed to provide flowing parallel sample and reference signals. The chromatograms were recorded on a Varian Model A-25 Recorder in the case of the Refractive Index detector and on a Linear Instru-

ments Model 252A, integrating strip chart recorder in the case of the U.V. detector. The U.V. detector was a 254 nm fixed wavelength detector.

The columns which were used were a 15 cm long, 4.6 mm i.d. Supelcosil LC 18 bonded phase column (Supelco, Inc.), packed with an 18 carbon straight chain bonded phase packing bonded to a 5 micron spherical silica; and an Alltech Associates column, 25 cm long, 4.6 mm i.d., packed with 10 micron Bondapak Phenyl (Waters Associates) phenyl bonded phase packing. Both columns achieved comparable separation and retention and either one could probably be used for the analysis. However, the major portion of the work described here was carried out using the phenyl bonded phase column.

The mobile phase used for this study was an aqueous phosphate buffer prepared from 85% phosphoric acid (Fisher Scientific, ACS Certified Reagent) and NaOH (Fisher Certified Reagent). The buffer system was made 0.06 molar in total phosphate, adjusted to pH2.00 with base, and degassed prior to use. Buffer degassing was accomplished by stirring the solution vigorously while under aspirator vacuum for at least one full hour. The degassed buffer was then transferred to the HPLC solvent reservoirs, taking care to minimize time of exposure to air, and then used without any further check of pH. No apparent difference in results was seen when the buffer was prepared with either laboratory deionized water or with HPLC quality water that was purchased for this study. The ultra-pure HPLC water was used in order to avoid possible complications.

Discussion

High pressure liquid chromatographic separation of CP and CP amide has been tried, using both ion exchange and reverse phase methods,^{5,6} but little success has been achieved in either case. The difficulty apparently arises from attempting the separation on the materials in the cobalt (III) form. The two cobalt (III) compounds are quite similar in molecular size, geometry and overall charge, thus the chromatographic systems have difficulty in discriminating between the two complexes. A convenient way to circumvent these problems, and to simplify the separation, is to reduce the cobalt (III) complexes, according to reactions 1 and 2, prior to attempting the chromatographic separation. The problem thus becomes one of separating the organic heterocyclic compounds 5-cyanotetrazole and 5-carboxamidotetrazole instead of the cobalt (III) complexes. The two organic heterocyclic compounds are readily separable on a reverse phase column as is indicated by the well resolved peaks shown in Figure 2.

Figure 2: a: Aged partially decomposed 5-cyanotetrazole (Cordova Chemical Co., Lot 5). 5-carboxamidotetrazole decomposition product retention time 2.9 min, 5-cyanotetrazole 3.9 min.
 b: 5-cyanotetrazole (Cordova Chemical Co., Lot 5) purified by ether recrystallization. Trace 5-carboxamidotetrazole retention time 2.9 min, cyanotetrazole 4.1 min.
 c: Mixture of CP and CP amide reduced over Sn in HClO_4 . 5-carboxamidotetrazole retention time 2.9 min, 5-cyanotetrazole 4.4 min.

Detector was refractive index. Chromatographic column was Supelcosil LC-18.

The heterocyclic compounds can be separated on a reverse phase column by using an aqueous acidic phosphate buffer to enhance their organic character, as opposed to using organic solvents or mixed solvents which have not given satisfactory separation.⁷ The pK values for the 5-cyano- and 5-carboxamido-tetrazoles indicate that these compounds exhibit a fairly strong acidic character. The 5-carboxamidotetrazole with a pKa of 2.48 is a significantly stronger acid than acetic acid which has a pKa of 4.75 and the 5-cyanotetrazole is an even stronger acid yet. Because of their strong acidity the two tetrazoles should exhibit the equilibria shown in reactions 3 and 4

and when in neutral and/or alkaline media, the equilibria should lie far to the right. It is, therefore, necessary to use an acidic medium to suppress the ionization of the tetrazole compounds and thereby force the equilibria to the left. Allowing the equilibria to lie to the right would make the charged species predominate and the separation by reverse phase HPLC would be unsatisfactory since the charged species would not be retained by reverse phase columns. Furthermore, at alkaline pH the hydrolysis of the cyanotetrazole to carboxamidotetrazole tends to be enhanced³ and thus is likely to give erroneous results.

A pH of 2.00 was chosen for the phosphate buffer because the pK_a values of the tetrazoles indicate that at this pH the percentage of protonated 5-carboxamidotetrazole (75%/25%, acidic/basic) is considerably larger than the percentage of protonated 5-cyanotetrazole (~33%/67%, acidic/basic). Thus, pH 2.00 provides a medium where the amount of reverse phase separable carboxamidotetrazole is enhanced while that of 5-cyanotetrazole is suppressed. This is convenient because the CP amide is present in CP at levels of only ~3 weight percent; therefore, it is beneficial to minimize the amount of reverse phase active cyanotetrazole in order to minimize column saturation effects which could adversely affect the retention of 5-carboxamidotetrazole. If the pH is much lower than pH 2.00, the above saturation effects would probably destroy the utility of the separation. Use of a higher pH would result in deprotonation of both compounds and result in a loss of reverse phase active 5-carboxamidotetrazole, which is already at a rather low concentration.

The Zn/Hg system was chosen for the reduction because of considerations of reaction rate and cleanliness. Reduction with the Zn/Hg system is relatively fast and does not appear to produce any interfering products. The same conditions for reduction were tried using tin as the reductant. However, the system was rejected due to complications arising from incompatibility of Sn^{2+} and phosphate buffer. When Sn^{2+} was added to phosphate buffer a precipitate was formed which was believed to be a tin phosphate compound; therefore, the Sn system was abandoned because of concern that the precipitate might cause blockage of the HPLC column and/or plumbing.

Experiments have shown that reduction using Zn/Hg reduces the Co (III) compounds with no adverse effects on the heterocyclic ligands, at least not in the time frame of the analysis. Treatment of both 5-cyanotetrazole and 5-carboxamidotetrazole in acidic media over Zn/Hg in the same manner that CP and CP amide were reduced gave no indication that either compound was affected. The chromatograms for the treated and untreated ligands were virtually identical, Figure 3; no measurable amount of 5-carboxamidotetrazole was lost due to the treatment nor was any generated from the 5-cyanotetrazole when it was similarly treated. Figure 3 shows the results for 5-carboxamidotetrazole. No signal was obtained for 5-cyanotetrazole at a detector wavelength of 254 nm.

FIGURE 3 a: 5-carboxamidotetrazole (3.89×10^{-4} M) dissolved in 0.45 M HClO_4 and aged 40 minutes, then chromatographed on a phenyl bonded phase reverse phase column. Retention time, 4.82 minutes.
 b: 5-carboxamidotetrazole (3.89×10^{-4} M) dissolved in 0.45 M HClO_4 and stirred over Zn/Hg for 40 minutes, then chromatographed on a phenyl bonded phase reverse phase column. Retention time, 4.82 minutes.

Verification that the tetrazole compounds were indeed reverse phase separable was obtained using refractive index detection, because the cyanotetrazole does not absorb in the U.V. at 254 nm, Figure 4. However, because the U.V. detector is a more sensitive and stable detector it was used as the analytical detector for the 5-carboxamidotetrazole. It should be pointed out that the detector used was a fixed wavelength 254 nm detector and that, according to Figure 4, use of a variable wavelength detector at a lower wavelength might afford a sensitivity enhancement of as much as a hundred-fold for 5-carboxamidotetrazole.

FIGURE 4. Ultraviolet absorption spectra of 5-cyanotetrazole and 5-carboxamidotetrazole in aqueous media.

The results for CP amide contaminant as obtained directly from the chromatograms are in terms of concentration and must be converted into terms of weight percent in order to be directly comparable to both the TLC and I.R. results. The conversion is carried out using equation 5,

$$\text{Wt. \% CP amide} = \frac{[\text{CP amide}]}{[\text{CP}]} \times \frac{\text{M.W. CP amide}}{\text{M.W. CP}} \times 100 \quad (5)$$

where $[\text{CP amide}] \equiv$ concentration of CP amide as determined from the chromatogram and $[\text{CP}]$ is calculated assuming that the entire sample weight used in the Zn/Hg reduction is pure CP. In this paper, brackets will be used to signify molar concentrations.

Results

The results of the present study indicate that High Pressure Liquid Chromatography is a viable alternative to both the thin layer chromatographic and the infrared methods of analysis. Comparison of the results obtained by all three methods, Figure 5 and Table 1, shows that, in those cases where it was possible to obtain data for all three methods, the HPLC method is in agreement with both of the other methods. The I.R. data were available for only a limited number of CP lots and in most cases CP from these lots was not available for HPLC analysis. The agreement with published TLC results is quite reasonable and, considering that the TLC results are only semi-quantitative, the differences obtained should not be considered significant. The rather large relative error associated with the analysis of CP lot EL 36353 is not

FIGURE 5: HPLC analysis of several lots of CP, O indicates CP lots, ● indicates CP amide standards used to calibrate the chromatograph. CP amide standard used was Lot EL41054. Dashed lines indicate confidence interval for standards at 99% confidence. Samples were reduced over Zn/Hg prior to being chromatographed on a phenyl bonded phase reverse phase column.

altogether surprising. Figure 4 shows that at 254 nm the detector is monitoring at a very weak portion of the 5-carboxamidotetrazole absorption spectrum. Thus, it is not surprising to see a certain degree of insensitivity in the detection of the carboxamidotetrazole and that the scatter in the data is more pronounced at the lower levels of carboxamidotetrazole concentration. Even so, Figure 5 indicates that within the confidence interval of the amide

TABLE 1.

Comparison of Weight Percent CP Amide Contaminant
in Various Lots of CP as Determined by Three
Different Methods.

CP	Wt. % CP Amide by HPLC, @ 99% Confidence	Wt. % CP Amide by TLC	Wt. % CP Amide by I.R.
EL 36353	0.86 ± 0.58	1.0	$0.98 \pm 0.01^*$
EL 41074	1.60 ± 0.11	2.0	1.40 ± 0.08
EL 43736-1	3.41 ± 0.09	5.0	
EL 36164-2	10.37 ± 0.11	9.0	

* This analysis is actually for EL 36353A, which is simply
the finest particles from Lot EL 36353.

standards the detector response to the 5-carboxamidotetrazole
is linear with a nearly zero intercept. Figure 6 shows
typical chromatograms of the various CP lots analyzed.

Figure 7 and Table 2 show the analysis results of CP
samples to which CP amide has been deliberately added (CP lot
EL 36353 was used, because it was the purest of the CP
available). The results obtained are again in reasonably
good agreement with results calculated from the weight per-
cent of CP amide added. The sample with zero weight percent

FIGURE 6. Chromatograms of various lots of CP, reduced over Zn/Hg and separated on a 10 μ m Bonda pak phenyl reverse phase column; mobile phase was 0.06 M Na phosphate buffer at pH 2.03.

a: Lot El-36353, 4.81×10^{-3} M;

b: Lot El-41074, 4.81×10^{-3} M;

c: Lot El-43736-1, 4.83×10^{-3} M;

d: Lot El-36164-2, 4.83×10^{-3} M.

Retention time is 4.75 ± 0.02 minutes.

*: This peak is unidentified.

CP amide added is in essence a repeat of the analysis of CP Lot EL 36353 which is listed in Table 1. The results obtained for EL 36353 in Table 2 are in excellent agreement with the results from both the TLC and infrared data listed in Table 1, and well within the error listed there for the previous analysis by HPLC.

FIGURE 7. HPLC analysis of CP (El 36353) to which varying amounts of CP amide (El 41054) have been added. ● indicates CP amide (El 41054) standards which have been used to calibrate the chromatograph. ○ indicates CP to which CP amide has been added. Samples were reduced over Zn/Hg, then chromatographed on phenyl bonded phase reverse phase column. Dashed lines indicate the confidence interval for the standards at 99% confidence.

TABLE 2.

Results of HPLC Analysis of CP* to which CP Amide has been added.

[CP Amide] Found by HPLC	[CP] Calculated from Total Wt. of Sample	Wt. % of CP Amide Found by HPLC	Wt. % of CP Amide Added to CP	Wt. % Found Corrected for CP Amide in CP* @ 99% Confidence
$(4.81 \pm 0.61) \times 10^{-5}$	5.00×10^{-3}	1.00 ± 0.13	0.00	0.00
$(9.28 \pm 0.53) \times 10^{-5}$	5.05×10^{-3}	1.91 ± 0.11	1.05	0.91 ± 0.17
$(2.08 \pm 0.12) \times 10^{-4}$	5.17×10^{-3}	4.18 ± 0.23	3.26	3.18 ± 0.26
$(2.93 \pm 0.06) \times 10^{-4}$	5.27×10^{-3}	5.80 ± 0.11	5.02	4.79 ± 0.17
$(3.71 \pm 0.12) \times 10^{-4}$	5.34×10^{-3}	7.24 ± 0.23	6.40	6.24 ± 0.26

* Lot EL 36353 was used because it is of the highest purity available.

There is a trend to the data found in Table 2 and that is that in all cases the weight percent of CP amide found, after correction for the contribution from EL 36353, is a bit lower than expected from the calculated weight % added. This leads to the conclusion that a systematic error is involved, and that the most probable source of the error is the CP amide added and not the technique itself. The CP amide complex is known to be hygroscopic and to take up water quite readily. Therefore, even though it was stored in a dessicator after moisture analysis was done, it is likely that it absorbed some water during its frequent and repeated handling outside the dessicator in the course of the procedure development. The original moisture analysis, which was actually a perchlorate analysis, gave a value of $42.1\% \text{ ClO}_4^-$ for CP amide Lot EL 41054, which corresponds to a molecular weight of 473.08 for the amide complex and reflects the presence of one water of hydration. The above trend only reflects the ease with which CP amide will absorb moisture and serves to point out that care should be taken when handling the standards for this technique.

Conclusions

The present work demonstrates the utility of High Pressure Liquid Chromatography as a tool for the analysis of CP amide in CP. The technique admittedly has the drawback that it is a somewhat indirect method requiring a prior reduction of the compounds of interest in order to do the analysis.

However, the reduction is a very straightforward reaction and the entire analysis can be carried out in a significantly shorter time than the infrared analysis, and with comparable results. With the proper precautions in handling the CP amide standards, to avoid moisture uptake, the technique should be a quite reliable method for doing the analysis of interest.

Addendum

In addition to the use of Sn as the reductor, a point which was discussed earlier, several other alternate media for carrying out the analysis were also tried. These included the use of hydrochloric, sulfuric, and phosphoric acids as the media for the reduction over Zn and the use of a sulfate buffer as the mobile phase in the chromatograph. Hydrochloric acid was rejected because chloride is known to corrode the stainless steel of which much of the chromatograph is constructed, the sulfuric acid appeared to reduce the detectability of 5-carboxamidotetrazole and the phosphoric acid gave a very fine dark precipitate of unknown identity when allowed to stand over the Zn/Hg -CP amide reaction solutions, thus all of these acids were rejected. The sulfate buffer was rejected because it was feared that it also would result in a lower sensitivity. The phosphate buffer was chosen because it is routinely used for chromatography and no adverse affects were expected or experienced with it.

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