



Original Research Article

A Green Approach for Photometric Determination of Copper β -Resorcyate in Double Base Solid Propellants

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ARTICLE INFO

Article history

Submitted: 2021-08-19

Revised: 2021-09-17

Accepted: 2021-10-11

Manuscript ID: CHEMM-2108-1366

Checked for Plagiarism: Yes

Language Editor:

Dr. Behrouz Jamalvandi

Editor who approved publication:

Dr. Zeinab Arzehgar

DOI: 10.22034/chemm.2021.138827

KEYWORDS

Copper β -resorcyate

Spectrophotometric determination

Micellar media

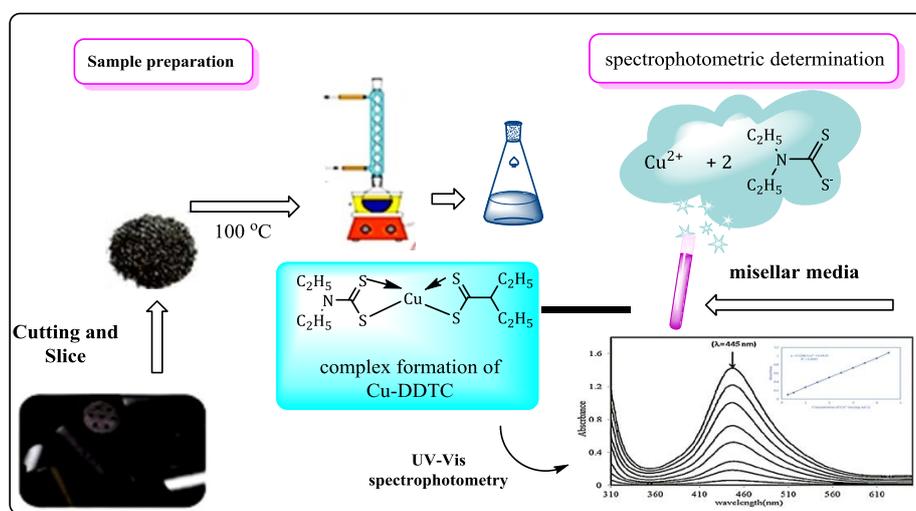
Double base Solid propellants

Sample preparation

ABSTRACT

This paper describes the applicability of micellar media instead of extraction steps with toxic solvents for direct determination of copper beta-resorcyate as burning rate catalyst in double base propellant (DB propellant). The method is based on a simple and safe sample preparation of DB propellant, and then complex formation of copper ion with Diethyldithiocarbamate (DDTC) in the presence of sodium dodecyl sulfate (SDS) as a micellar media. Under optimal conditions, at λ_{\max} = 445 nm, the calibration graph was linear in the range of 0.25-4.5 $\mu\text{g mL}^{-1}$ for copper with detection limits 0.125 $\mu\text{g mL}^{-1}$. The validity of the method was evaluated by means of the data statistical analysis. For this purpose, the method was applied to the determination of copper beta-resorcyate in DB propellant and the results were statistically compared based on t- and F-tests with those obtained by the by ICP-AES. There was no significant difference between the mean values and the precisions of the two methods at the 95% confidence level. The results showed that the proposed method offers an accuracy and reliable approach for the determination of copper β -resorcyate in DB propellant, and can be suggested as a routine method in military quality control laboratories.

GRAPHICAL ABSTRACT



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Introduction

The main goals of Green Analytical Chemistry (GAC) are eliminating the use of hazardous solvents that are hazardous to human health and the environment and thus reducing the amounts and toxicity of wastes and replacing them with benign ones, reducing sample treatment steps and consumption of organic solvents and increasing the safety of operators [1]. Considering the twelve principles of GAC and environmentally friendly aspects of these principles, factors like sample preparation, reagents, and instrumentation are very prominent in GAC. Sample preparation prior to analysis is one of the most important steps in many analytical protocols. Therefore, to make greening this stage is very important from the point of view of GAC. So, recently, sample preparation techniques focus on miniaturization, simplification and safety of methods [2].

Surfactants replacing organic solvents can be a major aspect in this process by both replacement of a solvent and a reduction in the amount of chemicals used. Because of their widespread use in home and industry, surfactants commonly find their way into the environment [3-5]. The evaluation of environmental risk assessment and biodegradability of these organic substances is an important consideration for public health and environmental impact. Besides, the research shows they are readily degradable under aerobic conditions. Surfactants can induce favorable shifts in equilibrium constants and spectral properties, and speed up reactions by means of micellar catalysis [6-8].

A DB propellant generally contains an energetic polymer, such as nitrocellulose (NC), plasticized into a gel by an energetic plasticizer, such as nitroglycerine (NG). Additives may be included in these double base propellants to improve the mechanical or ballistic properties of the propellant [9,10]. The double base solid propellants often require the use of ballistic modifiers to modify the ballistic properties and alters the inherently high dependence of the burning rate on chamber temperature and chamber pressure [11,12]. The burning rate-pressure relationship is one of the major criteria in the selection of propellants for any specific

application. To improve the operating conditions of an engine containing solid propellant, it is desirable that this rate of burning should remain substantially constant within a given pressure range, which is known as the plateau effect, and that the rate of burning should decrease in this pressure range, known as the mesa effect. Hence, the purpose of applying burning rate modifiers is to provide plateau or mesa burning at a desired range of pressure and burning rate [13,14]. Various organometallic salts and various oxides are known to modify the ballistic properties of double base solid propellants, and reported to produce pressure and temperature insensitivities, especially organometallic lead salts and copper salts, which are widely known to be effective. Compounds containing 2-hydroxybenzoic and 2,4-dihydroxybenzoic acids in various structural combinations with lead and copper are used as additives in certain DB propellants to modify ballistic properties [15,16]. Some of these salts, such as copper oxide (CuO) [17], copper chromite and copper β -resorcyate (β -Cu), have attracted a great deal of attention because of their high potential applications as catalysts and burning rate modifiers in the field of DB propellants [18]. As research shows that chemical stability and combustion rate in DB propellants are strongly influenced by (β -Cu). Based on the mentioned properties, it is necessary to determine copper β -resorcyate in double base solid propellant samples.

However, different methods are reported for determination of copper such as flame atomic absorption spectrometry [19,20], gas chromatography [21], spectrophotometric determination [22,23], among of these, the reported method in military standards, including military standard 286 for the determination of copper in DB propellants that involves the determination of copper by atomic absorption spectrometry (AAS) [24]. As the significant drawback of this method, the steps used are very long. On the other hand, sample preparation is done in hard digestion conditions by mineral acids such as perchloric acid and nitric acid. Due to the presence of compounds such as nitrocellulose and nitroglycerin in DB propellants, there can be a risk

of explosion Therefore, sample preparation and analysis of DB need special attention because hazardous materials are often used. As far as GAC principles are concerned, the development of novel environmentally friendly sample preparation process for DB propellant samples is necessary.

For routine quantitative analysis, a simple, rapid, user- friendly and economical technique is preferred. Spectrophotometric methods are the most commonly used techniques and attractive because of availability of the instrumentation, speed, precision and accuracy of the technique [25]. Diethyldithiocarbamate (DDTC) is one of the most widely reagent for routine colorimetric determination of copper ion. The complex of DDTC with Cu^{2+} ion is insoluble in water, so it requires solvent extraction prior determination [26]. Therefore, the possibilities toward green analysis in our study include a) eliminating organic solvents and extraction step by replacing of them with micellar media, b) reducing of hazardous material usage and miniaturization and simplification and safety of sample preparation steps, and c) evaluating the applicability of UV-Vis spectrophotometric technique as a simple, selective and user- friendly method for direct determination of copper β -resorcylate in DB propellants. According to our literature survey, this is the first report for spectrophotometric determination of copper β -resorcylate in DB propellants.

Material and methods

All chemical used in this study were analytically graded and used without any purification. Copper nitrate trihydrate, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (99.99%) was provided from (Sigma Aldrich), glacial acetic acid (Merck, 100%), sodium Diethyldithiocarbamate trihydrate (97%) was purchased from (Merk); sodium dodecyl sulfate (Sigma Aldrich, 99%), Triton X-100 (Sigma Aldrich), Cetyltrimethylammonium bromide ($\geq 98\%$), ethylene diamine tetra acetic acid disodium salt dihydrate, EDTA (99-101%), ammonia solution 25% (w/w) were provided from (Sigma Aldrich); and DB propellant and copper β -resorcylate were gifted by Iran's defense industry.

For preparation of stock solution of Cu (II), a sufficient amount of copper nitrate trihydrate was weighed and transferred to 100 mL volumetric flask then dissolved and made up to the mark with double distilled water. It was used as a stock solution (1000 $\mu\text{g}/\text{mL}$). Standard solutions were prepared from stock solution with appropriate dilution. Sodium Diethyldithiocarbamate trihydrate reagent solution (580 μM) was prepared by dissolving 0.01 g of this salt in 100 mL double distilled water. Masking agent solution (EDTA) 0.5% (w/v) was prepared by dissolving of 0.5 g of ethylenediamine tetra acetic acid disodium salt dihydrate in 100 mL double distilled water. Sodium dodecyl sulphate (SDS) solution (0.5% (w/v)) was prepared by dissolving, and diluting appropriate amount of anionic surfactant in 100 mL water. Triton X-100 solution (0.5% (w/v)) was prepared by dissolving, and diluting appropriate amount of nonionic surfactant in 100 mL water. Cetyltrimethylammonium bromide (CTAB) (0.5% (w/v)) was prepared by dissolving, and diluting appropriate amount of cationic surfactant in 100 mL water. 0.2 mol L^{-1} ammonia solution was prepared from 25% (v/v) ammonia solution.

Apparatus

UV-Vis spectrophotometer Model 3310 from Hitachi Company (Japan) with 1cm quartz cells was used for all the absorption measurements. All spectral measurements were performed using a blank solution as a reference. Magnetic stirrer (Hei Tec-Hidolph) and thermostatically controlled water bath (Model Hh-S4) were applied. Inductively coupled plasma -atomic emission spectrometer (Perkin Elmer PE Optima 5300DV) was used for comparison and validation of the method.

Experimental Procedure

To determine copper β -resorcylate, under optimum conditions, aliquots of solutions containing of Cu (II) (so that final concentration would be in the range of 0.25- 4.5 $\mu\text{g mL}^{-1}$), 2 mL of 0.5% (w/v) SDS solution, 2 mL of 0.5% (w/v) EDTA solution, 1.5 mL DDTC as a reagent, and finally, 1.5 mL of 0.2 mol L^{-1} ammonia solution

were transferred into a series of 10 mL standard flasks and the mixture was shaken thoroughly at room temperature, followed by the formation of stable yellow color. The solutions were made up to the mark with double distilled water and was transferred to quartz cell for absorbance measurement at 445 nm against a reagent as blank as the reference.

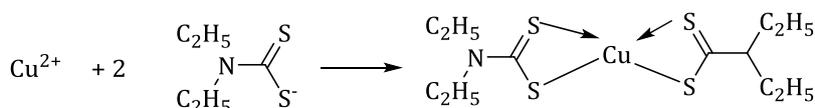
Sample preparation protocol of DB propellant for copper β -resorcyate determination

The DB propellant was cut into short, thin slices and for each test, a 1.0-2.0 g sample, weighed and solid to liquid extraction was used to prepare DB propellant for determination of copper β -resorcyate. The method involved adding 30 mL of acetic acid 6% (w/v) in two steps and refluxing on hot oil bath (100 °C) for two 45 min. The resulting solutions was cooled and carefully transferred into a 100 mL volumetric flask. The solution was made up to the mark with double-distilled water. Because, preparation of DB propellant was

performed in dilute acetic acid, nitroglycerin was dissolved and decomposed slowly and the explosion did not occur. Therefore, this is one of the steps in green analytical chemistry, in which sample preparation is performed in wet media without explosion.

Result and Dissection

In the SDS micellar and alkaline media, the complexation of Cu (II) with DDTC gives color product as shown in Scheme 1 cellular media strongly enhance the rate and equilibrium constants of the above reaction and the intensity of color of complex. Thus, it can be a successful method for spectrophotometric determination of Cu (II). Figure 1 shows the absorption spectra of the complexation product in the different concentrations of Cu (II), which exhibit a maximum absorbance at 445 nm. Therefore, all absorbance measurements were performed at this wavelength.



Scheme 1: complex formation of Cu-DDTC

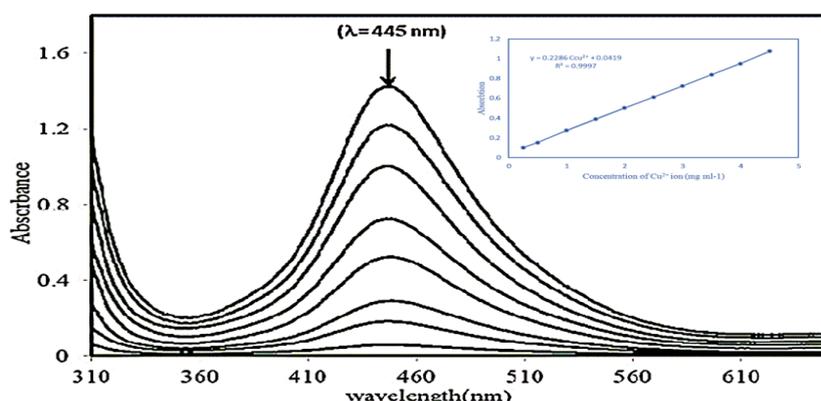


Figure 1: Absorption spectrum resulting from Cu^{2+} and DDTC reaction, conditions: concentration of Cu^{2+} : (0.25), (0.5), (1), (1.5), (2), (2.5), (3), (3.5), (4), (4.5) $\mu\text{g mL}^{-1}$, concentration of DDTC: 58 (μM), concentration of SDS: 0.1% (w/v), pH: 8.5, concentration of ammonia: 0.02 mol L^{-1} , temperature 25 °C, time: 1 min

Parameters affecting the process

In order to obtain a highly sensitive determination, the preliminary experiments were investigated and suitable ranges for the influencing parameters were found. These parameters were optimized by setting all parameters to be constant and optimizing one

each time. This optimization procedure may not lead to the actual optimum, although it certainly leads to an improvement of the analytical method. These parameters included the effect of concentration of reagent, the effect of surfactant type and its concentration, the effect of pH on reaction, time, and temperature on reaction.

Effect of DDTC concentration

The effect of DDTC concentration on the absorbance of the system was investigated within the range of 14.5- 116 μM . The results revealed that the absorbance increases by increasing the reagent concentration up to 58 μM , and does not change at higher concentrations (Figure 2), showing the reaction between Cu (II) and DDTC was complete in this concentration. Therefore, a concentration of 58 μM DDTC was applied in the proposed method.

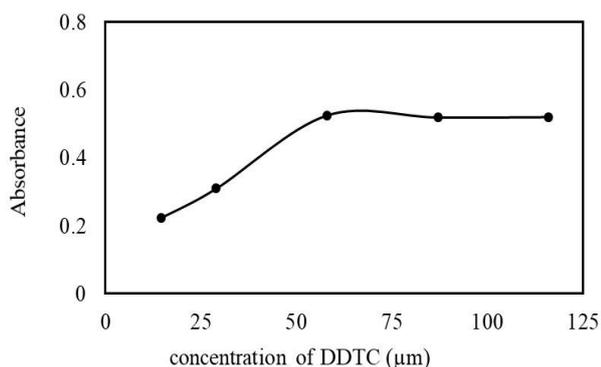


Figure 2: Effect of concentration of DDTC on reaction, conditions: concentration of Cu^{2+} : 2 $\mu\text{g mL}^{-1}$, concentration of SDS: 0.1% (w/v), pH: 8.5, concentration of ammonia: 0.02 mol L^{-1} , temperature 25 $^{\circ}\text{C}$, time: 1 min

Effect of the type and concentration of surfactants

Surfactants have a strong solubilizing power and also can enhance the absorbance characteristics of many absorbing species. The colored product of the copper reaction with DDTC reagent has low solubility in water. Therefore, sensitivity and absorbance of this complex is low in the absence of surfactant. In order to improve the sensitivity of system and solubility of complex formation, the effect of different surfactants was established. In order to investigate the effect of surfactant type some of surfactants such as Triton X-100 as nonionic surfactant, SDS as anionic surfactant and CTAB as cationic surfactant were studied. The results showed that in the presence of SDS, the solubility of complex in the water was high, the complex formation was fast, and the absorbance and the sensitivity of method were improved. Therefore, SDS was selected as micellar media for further investigation. The surfactant did not show any absorption in the absorption region of Cu-

DDTC and did not interfere with it. The effect of SDS concentration on reaction and sensitivity of method was investigated within the range of 0.01-0.15% (w/v). The result illustrated (Figure 3) a concentration of 0.1% (w/v) SDS showed maximum absorbance. Therefore, a concentration of 0.1% (w/v) SDS was applied in the proposed method.

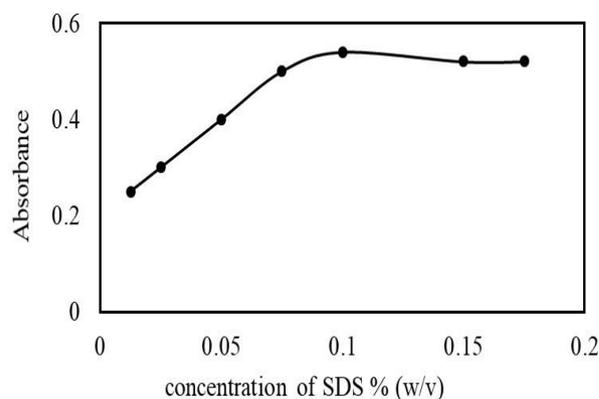


Figure 3: Effect of concentration of SDS on reaction, conditions: concentration of Cu^{2+} : 2 $\mu\text{g mL}^{-1}$, concentration of DDTC: 58 (μM), pH: 8.5, concentration of ammonia: 0.02 mol L^{-1} , temperature 25 $^{\circ}\text{C}$, time: 1 min

Effect of pH

The effect of pH on the formation of Cu-DDCT complex in the presence of SDS was investigated in the range 3-10. The pH varied by using 0.10 M HCl and 0.10 M ammonia solutions. It can be seen that the absorbance of the system increased to pH 8.5 and remained constant at higher pH, and at low pH, precipitation started. So, all subsequent studies were carried out at pH 8.5. So, the effect of ammonia concentration on the Cu-DDTC complex was studied in the range 0.005-0.045 mol L^{-1} . According to the results (Figure 4), a maximum increase was observed in the absorbance signal in the ammonia concentration of 0.02 mol L^{-1} . Thus, a concentration of 0.02 mol L^{-1} was chosen for the procedure.

Effect of temperature on sensitivity

Temperature is one of the parameters that can influence a chemical process. The dependency of the absorbance on equilibrium temperature was studied in the temperature range of 15-70 $^{\circ}\text{C}$. It was found that the absorbance remained constant in temperature range of 15-70 $^{\circ}\text{C}$. Therefore,

temperature has no effect on the reaction efficiency and this is a prominent feature of the method, which makes it a temperature-independent and simple method that can be done without needing to heating equipment.

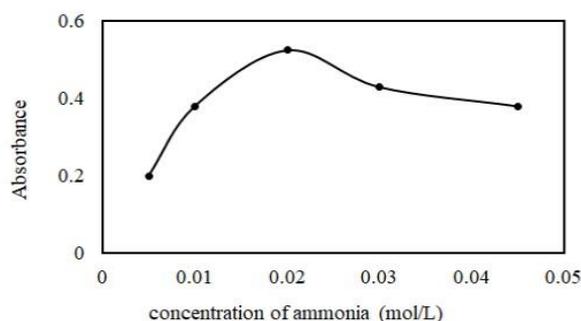


Figure 4: Effect of concentration of ammonia on reaction conditions: concentration of Cu^{2+} : $2 \mu\text{g mL}^{-1}$, concentration of DDTC: $58 \mu\text{M}$, concentration of SDS: 0.1% (w/v), pH: 8.5, temperature 25°C , time: 1 min

Study of stability of Cu-DDTC complex in micellar media

Stability of Cu-DDTC complex in micellar media with time was considered in the range of 1-20 min. The results showed that the reaction occurred immediately after the addition of the DDTC reagent and remained constant with time. It is concluded that the reaction of Cu^{2+} with DDTC reagent was rapid and the reaction time was not critical and it also indicates that by following the absorbance in different times, the absorbance values were constant, which shows that the Cu-DDTC complex is stable over time. The result of this study is that the method has good repeatability and there is an improvement in figures of merit.

Effect of ionic strength

The effect of ionic strength on the reaction stability and robustness of method was investigated by addition of different concentrations of salt solutions separately (NaCl, KCl, KNO_3 , Na_2SO_4 , KBr) into reaction solution in the range $0.15\text{--}0.75 \text{ mol L}^{-1}$. Based on the results, the change in ionic strength did not any affect absorbance of system and stability of Cu (II) - DDTC complex by increasing the electrolytes

concentrations in the range of $0.15\text{--}0.75 \text{ mol L}^{-1}$, so the results of this investigation were displayed that the ionic strength does not have any effect on the developed method. Further, this study showed that ionic strength in this range concentration had no effect on the absorption coefficient and peak displacement did not occur; therefore, no salt addition to the test solution was used for further studies. On the other hand, the analytical method proved to be robust since no statistically significant differences were found when samples were subjected to these solutions.

Analytical performance and method validation

In this work, we studied the reaction of Cu (II) and DDTC for determination of copper β - resorcyate, with the analytical characteristics of the optimized method, including regression equation, linear range, and limit of detection, repeatability and reproducibility. The linearity of the method was examined using nine concentration level solutions that were subjected to the developed method and it was found to be in the range $0.25\text{--}4.5 \mu\text{g mL}^{-1}$; the regression equation for proposed method is, $A = 0.2286C + 0.0419$, with a regression coefficient (r) of 0.9997 ($n = 10$) in which A is the absorbance at $\lambda_{\text{max}} = 445 \text{ nm}$. Limit of detection (LOD) and limit of quantification (LOQ) were $0.125 \mu\text{g mL}^{-1}$, $0.4 \mu\text{g mL}^{-1}$, respectively. The results showed that the proposed method has a high sensitivity range. The figures of merit of this study are presented in Table 1.

In order to investigate the intra-day precision of the proposed, triplicate absorbance measurements of the three reconstituted of prepared samples were done under the optimal working conditions and also the inter-day precision was determined by repeat measurements of three prepared samples over a 6-day period. The within-day precision revealed RSD by 2.5% and the considered inter-day precision was 2.6% . Intermediate precision was considered over 6 days by two analysts by using different spectrophotometric systems in three reconstitutions of prepared samples. The RSD value for of each precision analysis was less than 2.8% .

Table 1: Optical and statistical parameters of regression equation and validation parameters

Molar absorptivity, $\epsilon(\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1})$	1.45×10^4
Sandal's sensitivity ($\mu\text{g cm}^{-2}$)	4.37
Color of complex	Yellow
Measurement wavelength (nm)	445
Regression equation	$A = 0.2286C + 0.0419$
Regression coefficient (r^2)	0.9997
Slop of calibration curve	0.228
Linear range ($\mu\text{g/mL}$)	0.25-4.5
Limit of detection($\text{LOD} = 3s_0/m$)	$0.125 \mu\text{g mL}^{-1}$

Selectivity of method

A prominent feature of the proposed analytical method is the relative freedom of the method from the effect of interferences. To study the selectivity of the proposed methods, the effect of various ions on the determination of Cu^{2+} was tested under the optimum conditions. The tolerance limit was defined as the concentration of added ion causing less than $\pm 5\%$ relative error. The influence of some cations and anions (Na^+ , K^+ , NH_4^+ , Ca^{2+} , Pb^{2+} , Co^{2+} , Sn^{4+} , Mg^{2+} , Ti^{4+} , Cd^{2+} , Ni^{2+} , Fe^{2+} , Ca^{2+} , CO_3^{2-} , PO_4^{3-} , SO_4^{2-} , NO_3^- , Cl^- , Br^- , F^-) on determination of $2.0 \mu\text{g mL}^{-1} \text{Cu}^{2+}$ was studied according to the sample matrix and the selectivity of the proposed method was investigated. The results indicated that most of the species in tolerance ratio ($w_{\text{Species}}/w_{\text{Cu}^{2+}}$) 500:1 did not interfere with the determination of Cu (II). But Ca^{2+} , Pb^{2+} ions can interfere in determination of Cu^{2+} ions in tolerance ratio 50:1, so to remove the effects of interferences, EDTA solution 0.1% (w/v) was used as a masking agent. This indicates that under the mentioned reaction conditions, the formation of Cu(II)-DDTC complex is more favorable than the Cu(II)-EDTA complex, while these conditions will be more favorable for other ions (such as Ca^{2+} , Pb^{2+} ions) to form a complex with EDTA. Therefore, the interference effect of these ions will be removed with EDTA.

Application of method

The effectiveness of the proposed method was evaluated by determination of copper β -resorcylate in double base solid propellant samples. According to Table 2, the amount of copper resorcylate in the tested DB propellants was about 1.0 %, and also, according to the data in the patents, for example EP 0133 798 A2, the

amount of ballistic modifiers in DB propellants was about 0.5-10% [28]. The samples were prepared by using the mentioned method in sample preparation section. The results are presented in Table 4. The results obtained by the proposed method were confirmed by the measurements of Cu (II) content by ICP-AES methods (at $\lambda = 324.7$). The results were subjected to t-test and F-test (Table 2) and the data from these tests showed no significance differences between the spectrophotometric method and ICP-AES method at 95% confidence level. The comparison between the results obtained from the proposed method and the results of the ICP-AES method is a reason for the applicability of the proposed method for determination of copper β -resorcylate in double-base propellant samples. The results showed that the proposed method is suitable for determination of copper β -resorcylate in real samples.

In order to evaluate the applicability of developed method, it was applied to determination of CuR_2 in DB propellant samples prepared using the sample preparation section. The results of this investigation are presented in Table. 3. Spiking-recovery method was applied for investigation of accuracy and matrix effect of developed method. The recovery values were calculated according to the recovery equation 1:

$$\%R = \frac{C_1 - C_2}{C_3} \quad 1$$

In which C_1 is spiked portion, C_2 unspiked portion, and C_3 is concentration of analytes that add to spiked portion. According to the presented result in Table. 3, the recovery values for proposed method were in the range 98-105%. Good recoveries confirmed that the proposed method was not susceptible to the effect of matrix and the

method had high potential for application in complex formulation of DB propellants.

Table 2: Determination of copper and in DB propellant by proposed method and comparison with ICP-AES method

DB propellants	Proposed method		ICP-AES method		t-value (4.3)	F-value (9.28)	Determination of CuR ₂ in DB solid propellant		
	Cu (II) % Found in DB propellant	RSD%	Found Cu (II) % in sample	RSD%			Composition of CuR ₂ (%) in DB propellants	Found% in DB propellant	Recovery (%)
propellant 1	0.16 ± 0.005	3.125	0.158 ± 0.006	3.79	1.73	1.44	0.9	0.92 ± 0.01	102.22
propellant 2	0.18 ± 0.004	2.22	0.178 ± 0.005	2.80	1.15	1.59	1.0	1.05 ± 0.05	105

^a Calculated value

^b Theoretical value based on the paired t-test at the level of significance of p = 0.05

Table 3: Results of analysis of CuR₂, in DB propellant

Analytes	Spiked (µg mL ⁻¹)	Found (µg mL ⁻¹) ± SD	Recovery%
CuR ₂	0.0	2.73 ± 0.075	-
	5	7.68 ± 0.20	98
	10	12.88 ± 0.25	105
	20	23.0 ± 0.35	102

Conclusion

Our investigation revealed that the proposed method is simple, very sensitive and user-friendly spectrophotometric procedure for determination of copper β -resorcylate in a wide dynamic range that can be applied to DB propellants with satisfactory recoveries. In comparison to ICP-AES method, this method is routine with low instrumentation and operation cost. Due to the use of surfactant to improve sensitivity and elimination, the use of toxic solvents and short time of analysis, the proposed method can be considered as a green method in accordance with green analytical chemistry (GAC) principles. Method validation shows that the method has high accuracy and precision and is robust. To the best of our knowledge, this is the first report on the determination of copper β -resorcylate in solid propellant samples and due to the fact that so far no method has been reported for determination of copper β -resorcylate as a burning rate modifier in DB propellants in military standards, this report can be proposed as a reliable method with high accuracy in military standards such as military standard 286 (MIL-STD-286) as a first report for determination of copper β -resorcylate in double base propellant samples. Also, the method proved to be suitable for analysis of routine quality

control of DB formulations and can be extended for further study in this field.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose.

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HOW TO CITE THIS ARTICLE

Ali Reza Zarei, Kobra Mardi. A Green Approach for Photometric Determination of Copper β -Resorcylate in Double Base Solid Propellants, *Chem. Methodol.*, 2021, 5(6) 513-521
 DOI: 10.22034/chemm.2021.138827
 URL: http://www.chemmethod.com/article_138827.html