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Utility of solid phase extraction for spectrophotometric determination of gold in water, jewel and ore samples

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ABSTRACT

A highly sensitive, selective and rapid method for the determination $\mu g\,L^{-1}$ level of Au(III) based on the rapid reaction of Au(III) with 2,3-dichloro-6-(3-carboxy-2-hydroxy-1-naphthylazo)quinoxaline (DCHNAQ) and the solid phase extraction of the colored complex with a reversed phase polymer-based C18 cartridge have been developed. The DCHNAQ reacted with Au(III) to form a violet complex of a molar ratio 3:1 [DCHNAQ to Au(III)] in the presence of 5.0 M of phosphoric acid solution and Triton X-100 medium. This complex was enriched by the solid phase extraction with a polymer-based C18 cartridge. The enrichment factor of 100 was achieved. The molar absorptivity of the complex is $2.73 \times 10^5\,l\,mol^{-1}\,cm^{-1}$ at 633 nm in the measured solution. The system obeys Beer's law in the range of 0.02–1.30 $\mu g\,ml^{-1}$, whereas the optimum concentration ranges obtained from Ringbom plot was 0.08–1.24 $\mu g\,ml^{-1}$. The relative standard deviation for ten replicates sample of 0.6 $\mu g\,ml^{-1}$ level is 1.28%. The detection and quantification limits, are 6.1 and 19.5 $ng\,ml^{-1}$ in the original sample. This method was applied to the determination of gold in water, jewel and ore samples with good results comparing to the GFAAS method.

1. Introduction

Gold is widely distributed in nature and the chemistry of gold remains an active research area [1]. Due to the low level of gold in the environmental samples and its great importance, many articles have been reported on the separation of traces of gold in water and other matrices containing Cd^{2+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, Mn^{2+}, Co^{2+}, Pd^{2+}, Hg^{2+}, Pb^{2+}, Pt^{4+}, Fe^{3+}, alkaline and alkaline earth ions before their actual determination [2–8]. However, the low level of gold in drinking waters is not compatible with the detection limit and some of these methods are expensive, unselective and require careful experimental conditions and considerable time consuming. Thus, preconcentration and separation techniques using liquid–liquid and liquid–solid are frequently required to improve the detection capability and the selectivity of these techniques [6,9–11].

A geological sample is a complicated analytical system. It has been found that the content of gold in geological samples is very low, and its coexisting elements are very rich. For the determination of trace levels of gold in geological samples, sensitive methods are frequently required. Many methods to determine gold have been reported, such as flame atomic absorption spectrometry (FAAS) [12], graphite furnace (GFAAS) or electrothermal atomic absorption spectrometry (ETAAS) [13], inductively coupled plasma mass spectrometry [14], and spectrophotometry [15]. Because the procedures of the sensitive analysis methods mentioned above are generally subject to the effect of a number of interferences, suitable preconcentration and effective separation processes are frequently required, such as the use of solvent extraction [12,16], active carbon adsorption [17], polyurethane foam concentration [18], solid-phase extraction [19], and complex-forming resin concentration [20]. These techniques are effective when used for off-line preconcentration and separation. However, it is inconvenient to use these techniques because of manual operation.

Gold is generally determined using various complexing and chromogenic agents such as diethyldithiocarbamate [21], 8-mercaptoquinoline [22], 2-quinolylaldoxime [23], thiomides [24], aniline [25], ethylxanthate [26], 2-pyridinealdoxime [27], anthranilic acid [28], di-2-thienylketoxime [29], or chromophryozole [15]. In most procedures, the sensitivity is very poor and the colour fades after a few minutes. In some instances the complex is a water-insoluble compound and requires extraction with organic solvents.

However, the routine spectrophotometric methods are often not sensitive enough to determine low concentrations of gold ion, the gold concentrations of only $\mu g\,L^{-1}$ or $ng\,L^{-1}$ level. For the determination of low concentrations of gold ion, a preconcentration step is usually required. Solid phase extraction is an attractive technique because of its notable advantages (such as higher enrich-

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ment factor, reduced contamination of the environment and the ability for rapid and simultaneous preparation of large sample quantities) [30–34]. In this paper, the solid phase extraction of Au–DCHNAQ complex with a reversed phase polymer-based C18 cartridge was studied. The polymer-based C18 is manufactured from a hydrophilic methacrylate polymer, which is functionalized with C18 ligands. It is a reversed-phase solid phase cartridge that provides a broad range of solvent choices and a pH range from 0.0 to 14. By using the polymer-based C18 cartridge, the Au–DCHNAQ complex was enriched by solid phase extraction in phosphoric acid medium and the enrichment factor of 100 was achieved. Based on this, a highly sensitive, selective and rapid method for the determination of Au(III) ions in water, jewel and ore samples was developed.

2. Experimental

2.1. Apparatus

A Perkin-Elmer Lambda 12 UV–vis spectrophotometer with a 10 mm quartz cell was used for all spectral measurements. The extraction was performed on a Waters solid phase extraction (SPE) device (that can prepare 20 samples simultaneously), and a reversed phase polymer C18 TM cartridge (methacrylate polymer functionalized with C18 ligands, 10 mm i.d., 15 mm, 30 μm particle) was obtained from Beijing Genosys Technologies, P.R. China. A Perkin Elmer atomic absorption spectrometry model *AAnalyst* 300 was used for all GFAAS measurements. An Orion research model 601 A/digital ionalyzer pH meter was used for checking the pH of solutions.

2.2. Reagent

Analytical reagent grade chemicals and doubly distilled water were used throughout. 2,3-Dichloro-6-(3-carboxy-2-hydroxy-1-naphthylazo)quinoxaline used in the present investigation was prepared according to the procedure described previously [35]. A 0.0423 g of DCHNAQ was dissolved in 100 ml of absolute ethanol (1×10^{-3} M). The solution was stable for more than one month.

A stock solution of gold(III) was prepared by dissolving a weighed portion of chemically pure $HAuCl_4 \cdot 4H_2O$ in 1.0 M HCl. The concentration of gold (1.56 mg ml^{-1}) was determined by titrimetry using the known procedure [36]. A working solution containing 10 μg ml^{-1} was prepared by appropriate dilution with distilled water. A 5.0 M solution of phosphoric acid was used. Triton X-100 solution (5.0%, v/v) was prepared by dissolving Triton X-100 with water.

2.3. General procedure

To a standard or sample solution containing no more than 1.3 μg of Au(III) in a 100 ml of measuring flask, 4.0 ml of 5.0 M of phosphoric acid solution, 2.0 ml of 1×10^{-3} M DCHNAQ solution and 2.5 ml of 5.0% Triton X-100 solution were added. The mixture was diluted to volume of 100 ml and mixed well. After 5.0 min, the solution was passed through the polymer based C18 cartridge at a flow rate of 20 ml min^{-1}. After the enrichment was finished, the retained complex is eluted from the cartridge at a flow rate of 5.0 ml min^{-1} with 1.0 ml of isopentyl alcohol in the reverse direction. The eluent was adjusted to the accurate volume of 1.0 ml in a 1.0 ml calibrated flask by adding micro amounts of isopentyl alcohol with a 200 μl syringe. The absorbance of this solution was measured at 633 nm in a 10 mm microcell against a reagent blank prepared in a similar way without gold.

2.4. Determination of gold in ores

A suitable weight of sample was dissolved in aqua regia, add 4.0 ml of 1:1 (v/v) sulphuric acid, and evaporate to sulphur trioxide fumes. Cool, neutralize, and adjust the hydrochloric acid concentration to 1.5 M. The gold content was extracted with three successive 10-ml portions of ether. The combined ether extracts was evaporated to dryness and the residue was dissolved in 0.4 M hydrochloric acid. The gold was determined by the general and the reference GFAAS methods.

2.5. Pretreatment and determination of gold in jewel samples

The jewel samples were first washed with ethanol to remove fat and dirt. When dry, an accurately weighed amount of ca. 0.5 g sample turnings were transferred to a beaker and 20 ml of aqua regia were added. The beaker was covered and heated gently by a thermostated hot-plate until the dissolution of the sample. When the dissolution of the sample was completed, the resulting solution was carefully evaporated to a small volume (5.0–7.0 ml) to remove NO_3^- ions. After cooling at room temperature, the resulting solution was diluted to 100 ml in a volumetric flask with Milli-Q water, adjusting its acidity to pH 0.8 with HCl. Furthermore, 0.25 ml of the resulting solution was diluted to 250 ml in a volumetric flask with Milli-Q water to bring the sample within the dynamic range of the calibration graph, adjusting its final acidity to pH 0.8 by HCl. Finally, the sample was analyzed using both the above-general procedure for aqueous solutions and the reference FAAS method, after filtration through a 45 μm filter.

3. Results and discussion

3.1. Absorption spectra

The absorption spectra of DCHNAQ and its Au(III) complex are shown in Fig. 1. The absorption bands of DCHNAQ and its complex in isopentyl alcohol medium are located at 527 nm and at 633 nm, respectively.

3.2. Effect of acidity

Results showed that the optimal conditions for the reaction of Au(III) with DCHNAQ is in an acid medium. Therefore, the effects

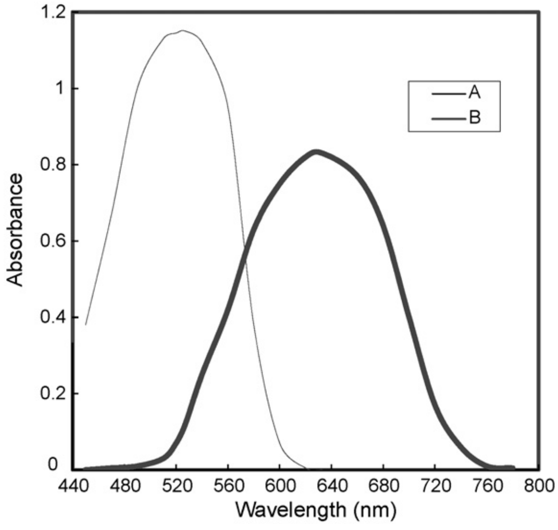

Fig. 1. Absorption spectra of (A) 2.0 ml 1×10^{-3} M DCHNAQ solution and (B) its complex with 0.6 μg ml^{-1} of Au(III) after SPE.

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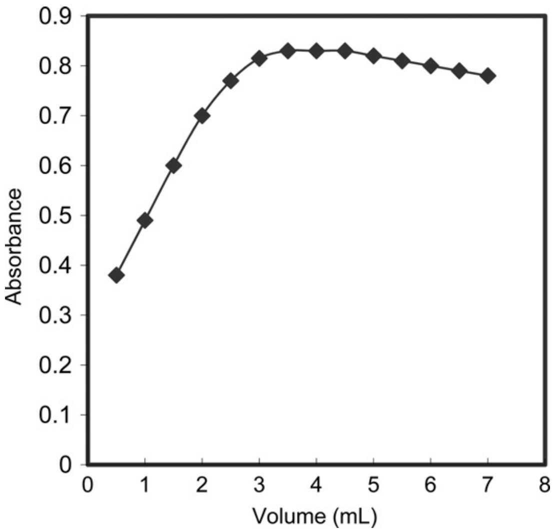

Fig. 2. Effect of the amount of 5.0 M phosphoric acid on the 0.6 μg Au(III) complexed with DCHNAQ.

of hydrochloric acid, perchloric, nitric, sulphuric, and phosphoric acids, on the color reaction of Au(III) with DCHNAQ were studied. Experiments show that 5.0 M phosphoric acid has the best effect, and the amount of 3.5–4.5 ml was found to give a maximum and constant absorbance (Fig. 2), so 4.0 ml of 5.0 M phosphoric acid was recommended.

3.3. Effect of surfactants

The effects of surfactants on the Au(III)–DCHNAQ system were investigated. The results showed that, in the absence of surfactants, anionic or cationic surfactants, the Au(III)–DCHNAQ chromogenic system gives a low absorption, whereas in the presence of nonionic surfactants, the absorption of the chromogenic system increases markedly. Various nonionic surfactants enhance the absorbance in the following sequence: Triton X-100 > emulsifier-OP > Tween-80 > Tween-60 > Tween-20. Accordingly, the Triton X-100 was the best additive, and the use of 2.0–3.0 ml of 5.0% Triton X-100 solution gave a constant and maximum absorbance (Fig. 3). Consequently, the use of 2.5 ml was recommended.

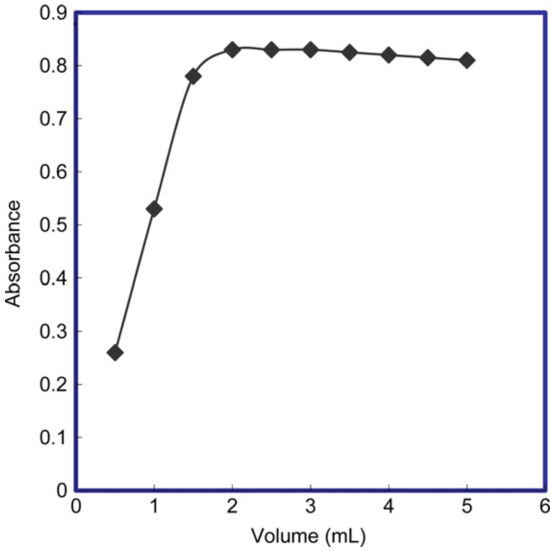

Fig. 3. Effect of 5.0% Triton X-100 on 0.6 μg Au(III) complexes with DCHNAQ.

3.4. Effect of DCHNAQ concentration

For up to 1.3 μg of Au(III), the use of 2.0 ml of 10^{-3} M of DCHNAQ solution was found to be sufficient for a complete reaction. Accordingly, 2.0 ml of DCHNAQ solution were added in all further measurements.

3.5. Stability of the chromogenic system

After the components are mixed, the absorbance reaches its maximum within 5.0 min at room temperature and remains stable for at least 6.0 h. After having been extracted into the isopentyl alcohol medium, the complex was stable for at least 12 h.

3.6. Solid phase extraction

Both the enrichment and the elution were carried out on a Waters SPE device. The flow rate was set to 20 ml min^{-1} for enrichment and 5.0 ml min^{-1} for elution.

Some experiments were carried out in order to investigate the retention of DCHNAQ and its Au(III) complex on the cartridge. It was found that the DCHNAQ and its Au(III) complex are retained on the cartridge quantitatively when they pass the cartridge as phosphoric acid medium. The capacity of the cartridge was determined as 42 mg for Au(III)–DCHNAQ complex in 100 ml of solution. In this experiment, the maximum amount of gold is only 1.3 μg. Therefore, the cartridge has adequate capacity to enrich the Au(III)–DCHNAQ complex.

In order to choose a proper eluent for the retained DCHNAQ and its Au(III) complex. We studied various organic solvents. For eluting the Au(III)–DCHNAQ complex from the cartridge, the volume of the solvent needed is 0.8 ml for isopentyl alcohol, 1.5 ml for DMF, 1.8 ml for acetone, 2.0 ml for acetonitrile, 2.2 ml for ethanol, and 2.5 ml for methanol. The maximum enrichment was achieved when isopentyl alcohol was selected as eluent. The experiment shows that it was easier to elute the retained DCHNAQ and its Au(III) complex in reverse direction than in forward direction, so it is necessary to reverse the cartridge during elution. Since 1.0 ml of isopentyl alcohol was sufficient to elute the DCHNAQ and its Au(III) complex from cartridge at a flow rate of 5.0 ml min^{-1}, the volume of 1.0 ml was selected.

3.7. Calibration curve and sensitivity

The calibration curve showed that the system obeys Beer's law in the concentration range of 0.02–1.3 μg Au(III) per ml in the measured solution. For more Accurate results, Ringbom optimum concentration ranges was found to be 0.08–1.24 μg Au(III) per ml in the measured solution. The linear regression equation obtained was $A = 1.3854C$ (μg ml^{-1}) + 0.0137 ($r = 0.9996$). The molar absorptivity was calculated to be 2.73×10^5 l mol^{-1} cm^{-1} at 633 nm, whereas Sandell sensitivity was found as 0.072 ng cm^{-2}.

The standard deviations of the absorbance measurements were calculated from a series of 13 blank solution. The limits of detection ($K = 3$) and of quantification ($K = 10$) of the method were established [37] and recorded in Table 1, according to the IUPAC definitions ($C_1 = KS_o/s$ where C_1 is the limit of detection, S_o is the standard error of blank, s is the slope of the standard curve and K is the constant related to the confidence interval. The relative standard deviation was 1.28% obtained from a series of 10 standards each containing 0.6 μg ml^{-1} of Au(III).

The sensitivity expressed as molar absorptivity of the proposed method is compared with those of published spectrophotometric methods (Table 2). The higher sensitivity of the proposed method is notable, greater even than that of the SPE [8] that used 5-(2-hydroxy-5-nitrophenylazo)thiorhodanine. Also, the proposed

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Table 1
Analytical parameters.

Parameter	Value	Parameter	Value
Beer's law limit ($\mu g\,ml^{-1}$)	0.02–1.3	Regression equation[a]	
Ringbom optimum range ($\mu g\,ml^{-1}$)	0.05–1.24	Slope (b)	1.3854
Molar absorptivity ($l\,mol^{-1}\,cm^{-1}$)	2.73×10^5	Intercept (a)	0.0137
Sandell sensitivity ($ng\,cm^{-2}$)	0.072	Correlation coefficient (r)	0.9996
Detection limit ($ng\,ml^{-1}$)	6.1	RSD (%)	1.65
Quantification limit ($ng\,ml^{-1}$)	19.5	S stoichiometric ratio (L:M)	3:1

[a] $A = a + bC$, where C is the concentration of iron in $\mu g\,l^{-1}$.

Table 2
Comparison of the sensitivity of the methods for spectrophotometric determination of Au(III).

Reagent	λ_{max} (nm)	$\varepsilon \times 10^{-3}\,l\,mol^{-1}\,cm^{-1}$	Medium	Ref.
Mepazine hydrochloride	514	2.18	Aqueous	[39]
4,4′-Bis(dimethylamino)-thiobenzophenone	540	12	Aqueous	[40]
3,3-Dimethyl-2-[4-(N-methyl-Nchloroethyl) amido phenylethylene] indole	555	7.29	Benzene	[41]
Cyclopentan-spiro-2-(1-methyl-2′,4′-dithio)-S-triazine	435	128	Aqueous	[42]
5-(4-Sodium sulphonatephenylazo)-8-aminoquinoline	605	148	Aqueous	[43]
Chrompyrazol	580	7.5	Toluene	[44]
Brilliant green	643	101	Toluene	[45]
Crystal violet	615	106	Benzene	[46]
Ethyl violet	551	5.04	Benzene	[47]
Victoria blue 4R	608	5.5	Benzene	[48]
Methylene blue	657	108	Dichloroethane	[49]
Rhodamine B	555	9.7	Diisopropyl ether	[50]
Nile blue	595	271	Aqueous	[51]
5-(2-Hydroxy-5-nitrophenylazo)thiorhodanine	520	137	DMF	[8]
5-(4-Carboxylphenylazo)-8-aminoquinoline	608	91.1	Aqueous	[52]
5-(4-Carboxylphenylazo)-8-hydroxyquinoline	520	199	Ethanol	[53]
5-(p-Aminobenzylidene)-thiorhodanine	550	123	Emulsifier-OP	[54]
Sodium 8-aminoquinoline-5-azobenzene-4′-sulphonate	611	156	Ethanol	[55]
3,3′, 5,5′-Tetramethylbenzidine hydrochloride	458	11	Aqueous	[56]
Isothipendyl hydrochloride	512	21	Aqueous	[57]
Bis(salicylaldehyde)orthophenylenediamine	490	250	Triton X-100	[58]
2,3-Dichloro-6-(3-carboxy-2-hydroxy-1-naphthylazo)quinoxaline	633	273	Isopentyl alcohol	This work

method is more sensitive than other methods [39–58], that based on spectrophotometry (Table 2).

3.8. Stoichiometric ratio

The nature of the complex was established at the optimum conditions described above using the molar ratio and continuous variation methods. The plot of absorbance versus the molar ratio of DCHNAQ to Au(III), obtained by varying the DCHNAQ concentration, showed inflection at molar ratio 3.0, indicating presence of three DCHNAQ molecules in the formed complex. Moreover, the Job method showed a ratio of DCHNAQ to Au(III) = 3.0. Consequently, the results indicated that the stoichiometric ratio was (3:1) [DCHNAQ:Au(III)]. The conditional formation constant (log K), calculated using Harvey and Manning equation applying the data obtained from the above two methods, was found to be 9.88, whereas the true constant was 10.10.

3.9. Interference

The influence of foreign ions on the determination of Au(III) was examined using the general procedure. The tolerance limit was taken as the ratio of foreign ions to the Au(III) ion determined that causes a ±4.0% error in the absorbance value. The results are listed in Table 3. The result shows that most common ions do not interfere with the determination suggesting the highly selectivity of the proposed method.

3.10. Analytical applications

In order to confirm the applicability of the proposed method, it has been applied to the determination of microgram amounts of gold in water and in real samples. For water samples, the samples were acidified with phosphoric acid and filtrated through a 0.45 μm filter. The gold contents were analyzed according to the general procedure. An GFAAS method was used as a reference method and the results are also shown in Table 4.

The proposed method was successfully applied to the analysis of four real samples (two jewel and two ore samples). The accuracy was further validated by comparison of the results obtained by the proposed method with those obtained by the GFAAS as a reference method.

The performance of the proposed method was assessed by calculation of the t-value (for accuracy) and F-test (for precision) compared with GFAAS method [13]. The mean values were obtained in a Student's t- and F-tests at 95% confidence limits for five degrees of freedom [38]. The results showed that the calculated values (Table 4) did not exceed the theoretical values. A wider range

Table 3
Tolerance limits for the determination of 0.6 μg of Au(III) with DCHNAQ (relative error ±4.0%).

Foreign ions	Tolerance limit, μg
Na^+, K^+, Li^+, $NO_3{}^-$, $PO_4{}^{3-}$	25,000
Ca^{2+}, Mg^{2+}, Cl^-, $ClO_4{}^-$, CH_3COO^-	18,000
Ba^{2+}, Mn^{2+}, Sr^{2+}, $SO_4{}^{2-}$, $S_2O_3{}^{2-}$, $SiO_3{}^{2-}$	11,500
Ce^{4+}, $WO_4{}^{2-}$, $MoO_4{}^{2-}$, $UO_4{}^{2-}$	7000
Cd^{2+}, Zn^{2+}, Cu^{2+}, Co^{2+}, $Ni^{2+,}$	4500
Fe^{3+}, Cr^{3+}, Al^{3+}, $SnO_3{}^{2-}$,	3000
Ti^{4+}, Bi^{3+}, $VO_3{}^-$, $CrO_4{}^{2-}$, F^-,	1000
$Pb^{2+,}$ Fe^{2+}, Ru^{3+}, Bi^{3+}, Sb^{3+}, Th^{4+},	750
$OsO_5{}^{2-}$, $SeO_3{}^{2-}$, $TeO_3{}^{2-}$, Ag^+, I^-, Br^-	500
Ir^{4+}, Rh^{3+}, Ru^{3+}, Pd^{2+}, Pt^{4+}	180
Hg^{2+}, CN^-, SCN^-	100
La^{3+}, Sm^{3+}, Gd^{3+}	50
Be^{2+}, Sc^{3+}	25

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Table 4
Determination of Au(III) in water, Jewel and ore samples.

Sample	Added			Proposed Method		GFAAS method	
	Au(III)	Au(III)	*t*-value	*F*-test	RSD %	Au(III)	RSD %
River Nile water ($\mu g\,l^{-1}$)	–	5.3	1.17	2.78	1.46	5.2	1.68
	4.0	9.25	1.29	3.13	1.79	9.25	1.43
	12.0	17.35	–	3.27	1.32	17.25	1.59
Well water ($\mu g\,l^{-1}$)	–	3.8	1.34	3.46	1.56	3.85	1.75
	5.0	8.85	1.08		1.81	8.85	1.54
	15.0	18.7	1.20	2.93	1.38	18.80	1.66
Jewel sample 1 ($mg\,g^{-1}$)	–	76.5	1.44	3.69	1.74	76.65	1.92
	6.0	82.55		3.25	1.31	82.60	1.54
	18.0	94.55	1.26	3.07	1.62	94.60	1.78
Jewel sample 2 ($mg\,g^{-1}$)	–	87.2		2.85	1.88	87.35	1.56
	5.0	92.3	1.40	3.73	1.73	92.35	1.87
	10.0	97.3	1.52		1.62	97.40	1.72
Ore sample 1 ($g\,T^{-1}$)	–	0.53		4.11	1.95	0.525	1.76
	0.5	1.04		3.65	1.63	1.03	1.28
	1.0	1.54	1.37		1.57	1.53	1.39
Ore sample 2 ($g\,T^{-1}$)	–	1.38	1.69		1.83	1.37	1.35
	1.0	2.37		3.27	1.92	2.375	1.54
	2.0	3.39	1.23	2.91	1.73	2.38	1.44

[a] Average of six determinations. [b] Theoretical values for *t*- and *F*-values at 95% confidence level for five degrees of freedom are 2,57 and 5.05, respectively.

of determination, higher accuracy, more stability and less time consuming, shows the advantage of the proposed method over other method.

4. Conclusions

The proposed method has the following characteristics:

(1) DCHNAQ is one of the most easily prepared high purity, sensitive, and selective spectrophotometric reagent for gold determination. Molar absorptivity of the chelate was found to be up to $2.73 \times 10^5\,l\,mol^{-1}\,cm^{-1}$ at 633 nm in the measured solution. The higher sensitivity of the proposed method is notable, greater even than that of the SPE that used 5-(2-hydroxy-5-nitrophenylazo)thiorhodanine. Also, the proposed method is more sensitive than other methods, based on spectrophotometry.
(2) The detection and quantification limits, are 6.1 and 19.5 ng ml^{-1} in the original sample.
(3) Most common ions do not interfere with the determination suggesting the highly selectivity of the proposed method.
(4) Successful application of the proposed method to the determination of low levels of gold in water, jewel and ore samples was carried out with good results.

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