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Chemiluminescence analysis of menadione sodium bisulfite and analgin in pharmaceutical preparations and biological fluids

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Abstract

A novel chemiluminescence (CL) flow system for two sulfite-containing drugs, namely, menadione sodium bisulfite (MSB) and analgin is described. It is based on the weak chemiluminescence induced by the oxidation of sulfite group in drugs with dissolved oxygen in the presence of acidic Rh6G. Tween 80 surfactant micelles showed a strong enhancement effect on this weak chemiluminescence. For MSB analysis, online conversion of MSB in alkaline medium into sodium bisulfite was necessary, whereas analgin could be determined directly. The proposed method allowed the measurement of 0.05–50 μg ml^{-1} MSB and 0.05–10 μg ml^{-1} analgin. The limits of detection (3σ) were 0.01 μg ml^{-1} MSB and 0.003 μg ml^{-1} analgin. The method was applied satisfactorily to pharmaceutical preparations as well as biological fluids.

Keywords: Chemiluminescence; Menadione sodium bisulfite; Analgin; Pharmaceutical analysis; Flow-injection analysis

1. Introduction

Analytical methods applying chemiluminescence (CL) coupled with flow injection analysis (FIA) offer simple, cheap apparatus and rapid, reproducible means of detection. It has been extensively used for the determination of sulfur-containing substances in recent years and many analytical applications have appeared in the literature. Stauff and Jaeschke [1,2] reported a method for the determination of trace sulfur dioxide in the atmosphere with the aid of a photon counter based on the weak chemiluminescence produced by the oxidation of SO_2 in aqueous solution with either acidic permanganate, cerium(IV) sulfate or hydrogen peroxide. Yamada et al. [3] found that addition of riboflavin phosphate could greatly enhance the intensity of light, which allowed conventional detection equipment to be used. There has been increasing interest in searching for organic substances including fluorescent and non-

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fluorescent compounds as sensitizers for this purpose [4–8]. More recently, Baeyens et al. reported the use of cerium(IV) oxidant for the oxidation of some hydrosulfo-compounds and sulfonamide-compounds [9,10]. Potassium permanganate and cerium(IV) are typical oxidants among various oxidants (such as iodine [11] or hydrogen peroxide [12]) for the determination of sulfur-containing substances. One of the major disadvantages of using potassium permanganate as oxidant is that the concentration of the oxidant should be as low as possible, as its absorption band overlaps the emission band [4,5]. Overlapping of bands is minimized when Ce(IV) is used as an oxidant [5]. Unfortunately, the Ce(IV) system suffers from high background and expensive reagent consumption.

A less expensive alternative that can provide a fast and simple quantitative measurement of sulfur-containing substances is the FIA-CL method without using additional oxidant such as potassium permanganate or cerium(IV). Our previous work [13] showed that the oxidation of (bi)sulfite in acidic rhodamine dyes in the absence of oxidant such as Ce(IV) or potassium permanganate is accompanied by a weak CL. This weak CL could be greatly enhanced by an organized surfactant such as Tween 20 or 80. The possible mechanism of the CL reaction between dissolved oxygen and sulfite may be that dissolved oxygen with an $E°$ (O_2/H_2O) of 1.229 V can oxidize HSO_3^- to a hydrogensulfite radical $HSO_3^{\bullet}$ and then $HSO_3^{\bullet}$ radicals react to produce $S_2O_6^{2-}$. $S_2O_6^{2-}$ will cause the excited intermediate product SO_2^*. Energy is transferred from SO_2^* to fluorophore such as Rh6G. In order to extend the application of this new CL reaction, in this paper, two sulfite-containing drugs, namely, MSB (a potential anticancer drug and an effective dose in vitamin K deficiency [14–17]) and analgin [(2,3-dimethyl-5-oxo-1-phenyl-3-pyrazolin-4-yl) amino methanesulfonate, a therapeutic agent typically used as an analgesic, antipyretic and antispasmodic; in addition, it also forms the active constituent of other drugs] were tested based on the above mentioned CL reaction. It was discovered that analgin could be determined directly, whereas, conversion of MSB in strong alkaline medium into sodium bisulfite was necessary for MSB analysis. The optimized procedure allowed the determination of studied drugs in pharmaceutical preparations and biological fluids.

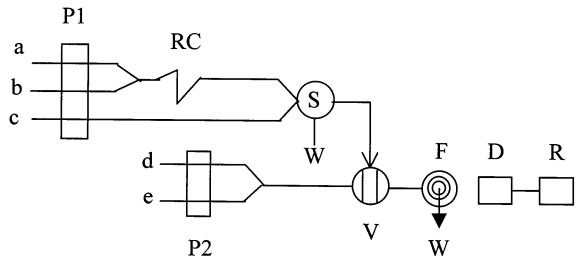

Fig. 1. Schematic diagram of the flow system for the determination of MSB and analgin. (a) 0.03 M Na_2CO_3; (b) MSB (sample or standard solution); (c) analgin (sample or standard solution); (d) rhodamine 6G in H_2SO_4; (e) Tween; S, switching valve; RC, reaction coil; V, injection valve; P1, P2, peristaltic pump; F, flow cell; W, waste; D, detector; R, recorder.

2. Experimental

2.1. Apparatus

All CL measurements were made by an R456 photomultiplier tube (Hamamatsu), which was operated at −850 V and placed close to the flow cell. The CL was transformed into an electrical signal and recorded with an XWT-204 recorder (Shanghai Dahua Instrument and Meter Plant).

A schematic diagram of the set-up is shown in Fig. 1. The flow system employed in this work consisted of two peristaltic pumps. One delivered the carrier stream (a mixture of Rh6G in H_2SO_4 and Tween 80) at a flow rate (per tube) of 3.0 ml min^{-1}. The other delivered a sample stream (in the case of analgin) or a sample stream and sodium carbonate (in the case of MSB) at a flow rate (per tube) of 2.5 ml min^{-1}. PTFE tubing (0.8 mm i.d.) was used to connect all components in the flow system. Sample solution (200 μl) was injected into the carrier stream by a six-way injection valve. S is a switching valve that allows switching between analgin determination and MSB analysis.

2.2. Reagents

All the reagents were of analytical-reagent grade unless specified otherwise; doubly distilled water was used for the preparation of solutions. MSB was obtained from Xi'an Chemical Reagent Company. Menadione sodium bisulfite solution was prepared daily by dissolving menadione sodium bisulfite in degassed water. MSB injections were obtained from a local hospital. Analgin was obtained from Xi'an Pharmaceutical Factory. A stock solution of 100 μg/ml analgin was prepared in 0.2 M KH_2PO_4–NaOH buffer (pH 7.8) daily. Analgin tablets were obtained from a local drug store. Tween 80 was obtained from Farco Chemical Supplies. Rhodamine 6G was obtained from Merck.

2.3. Procedure for determination of analgin

A series of working standard solutions with different concentrations between 0.05 and 10 μg/ml was prepared by diluting a concentrated fresh standard solution of analgin with KH_2PO_4–NaOH buffer. The CL signal was obtained by injecting 200 μl of the working standard solution or sample solution into the carrier (0.1 mmol/l Rh6G in 0.3 mol/l H_2SO_4 and 0.1% Tween solutions). The relative CL intensity versus analgin concentration was used for the calibration.

2.4. Procedure for determination of MSB

A portion of standard solution or sample solution and 0.03 M sodium carbonate were mixed and pumped into a 400-cm length of reaction coil (PTFE tubing, 1 mm i.d.) with a flow rate of 2.5 ml min^{-1}. The CL signal was measured by injecting 200 μl of online resulting solution into the carrier stream (0.08 mmol/l rhodamine 6G in 0.06 mol/l H_2SO_4 and 0.5% Tween solutions). The relative CL emission intensity versus MSB concentration was used for the calibration.

2.5. Procedure for analgin tablets

Ten tablets of analgin were weighed to obtain the mean tablet weight, then ground to homogenized powder and an accurately weighed portion powder corresponding to 50 mg was diluted in 0.2 M KH_2PO_4–NaOH buffer (pH 7.8) for the quantitative analysis.

2.6. Procedure for MSB injections

Injection samples, each with a nominal content of 4 mg of MSB in 1 ml, were diluted to 100 ml with doubly distilled water. A 2.5-ml aliquot of diluted solution was taken into a 100-ml flask, and made up to volume with doubly distilled water and then used for analysis.

2.7. Procedure for spiked urine and serum

Whole blood samples (10 ml, from local hospital) were centrifuged at 4000 rev./min for 20 min. The supernatant was aspired to a test tube and used as serum sample. Add an aliquot of standard aqueous solution of the studied drug to 0.2 ml of fresh urine or 0.2 ml of serum sample. After mixing for 2 min, it was deproteinized by adding 2 ml of 0.1 M $Ba(OH)_2$ and 1.8 ml of 0.1 M $ZnSO_4$. From this 4-ml volume, which was then centrifuged for 10 min at 4000 rev./min, 1 or 0.5 ml of the centrifugate was diluted to 100 ml with water or KH_2PO_4–NaOH buffer solution and then used for analysis. The absolute recovery was determined for each drug by comparing the representative CL intensity of the treated urine or serum samples with the CL intensity of the standard drug at the same concentration.

3. Results and discussion

3.1. Optimization of experimental variables

A series of experiments was conducted to establish the optimum analytical variables. The parameters optimized included reagent concentrations and some physical variables, including the flow rate and the length of reaction coil (in the case of MSB analysis). Table 1 shows the performance data for the determination of the studied drugs.

Table 1
Performance data for the CL determination of the studied drugs

Parameters	Analgin	MSB
Conc. of Rh6G (mM)	0.1	0.08
Conc. of H_2SO_4 (M)	0.3	0.06
Conc. of Tween (%)	0.1	0.5
Conc. of Na_2CO_3 (M)	–	0.03
Length of reaction coil (cm)	–	400
Linear calibration range (μg/ml)	0.05–10	0.05–50
Limit of detection (μg/ml)[a]	0.003	0.01
Correlation coefficient	0.9981 ($n=7$)	0.9999 ($n=8$)
Regression equation	$I=52.48C+9.77$	$I=21.07C+0.09$

[a] 3σ.

3.1.1. Effect of Tween 80 concentration

The concentration of Tween 80 was varied between 0 and 1% (V/V). Fig. 2 shows the effect of Tween concentration on the CL intensity of each drug. The greatest CL signal was obtained with 0.1 and 0.5% Tween for analgin and MSB, respectively. Upon reaching a certain minimum concentration (the critical micelle concentration, CMC, 13 μg ml^{-1} for Tween 80 [18]), amphiphilic surfactant molecules tend to form micelle. The local microenvironment in micelle media leads to a significant increase in CL quantum yield [19]. The compartmentalization due to the formation of micelle prevents oxygen-based quenching, leading to CL increase. On the other hand, micelle-volume contract due to the formation of micelle decreases the energy transfer efficiency from luminophore to energy transfer reagent. Due to two opposite actions, there must be a concentration at which the greatest compartmentalization and smallest micelle-volume contract obtained, lead to the largest CL increase. So the signals decreased at higher concentrations of Tween 80 after reaching the maximum.

3.1.2. Effect of Rh6G concentration

The rhodamine 6G concentration was varied in the range 0.01–1 mM in order to maximize the CL signal. Fig. 3 shows the effect of Rh6G concentration on the CL intensity of each drug. The maximum CL intensity was obtained with 0.1 mM, 0.08 mM Rh6G for analgin, MSB, respectively. At higher concentrations, the signal decreased sharply probably because of self-absorption of the emission by rhodamine 6G.

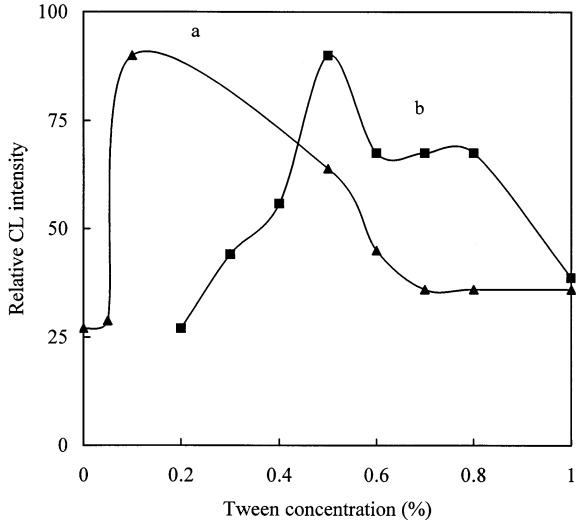

Fig. 2. The effect of Tween concentration. (a) Analgin; (b) MSB.

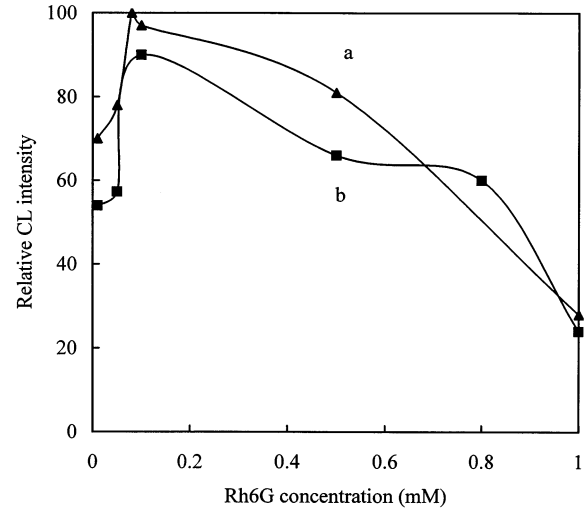

Fig. 3. The effect of Rh6G concentration. (a) Analgin; (b) MSB.

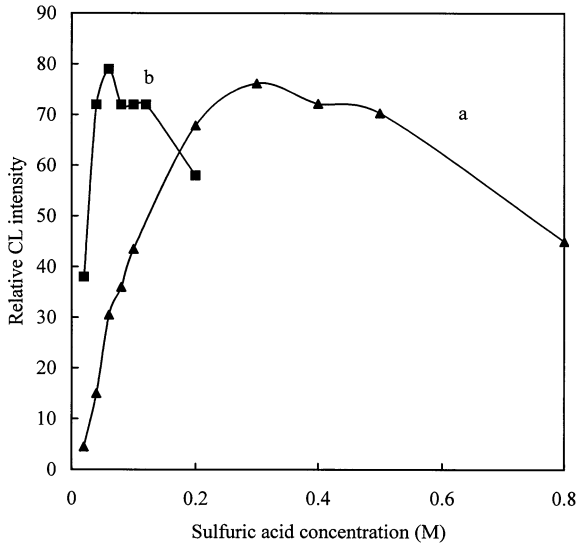

Fig. 4. The effect of sulfuric acid concentration. (a) Analgin; (b) MSB.

3.1.3. Effect of sulfuric acid concentration

The effect of sulfuric acid (which was added to the Rh6G solution) concentration was studied at different concentrations from 0.02 to 0.8 M. Fig. 4 shows the effect of sulfuric acid concentration on the CL intensity of each drug. The maximum CL intensity was obtained with 0.3 and 0.06 M sulfuric acid for analgin and MSB, respectively.

3.1.4. Effect of sodium carbonate concentration on MSB analysis

The effect of sodium carbonate was studied. In neutral and alkaline media, there is a reversible equilibrium reaction between MSB and sodium bisulfite [20,21] (Fig. 5). The equilibrium lies toward MSB at pH 6–10 and toward sodium bisulfite at pH 11 or above. Primary experiments showed that different bases such as sodium carbonate, sodium hydrate, sodium orthophosphate and sodium tetraborate at the same pH of 11.3 showed no relevant influence. Therefore, sodium carbonate was selected in this study. The effect of sodium carbonate concentration was studied in the range 0.01–0.1 M. The signal increased with increasing sodium carbonate concentration up to 0.03 M, but it decreased at higher concentrations. A 0.03 M sodium carbonate solution was selected.

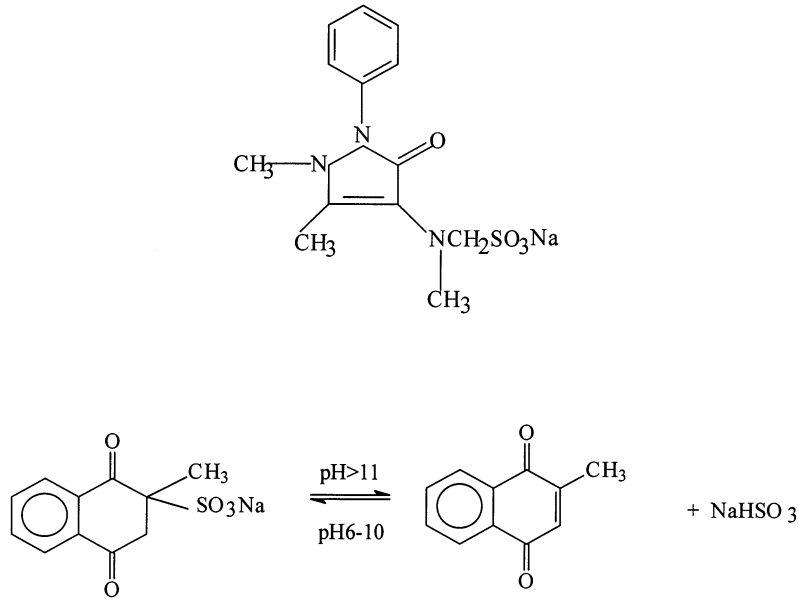

Fig. 5. Structure of analgin and reaction scheme for MSB in alkaline media.

Table 2
Tolerable concentration ratios with respect to studied drugs for some interfering species

Tolerable concentration ratio	Substances for MSB	Substances for analgin
1000	Mg^{2+}, Na^{+}, K^{+}, NH_4^{+}, Ca^{2+}, $C_2O_4^{2-}$, HCO_3^{-}, Cl^{-}, $H_2PO_4^{-}$, SO_4^{2-}, Zn^{2+}, acetate, glucose, fructose, sucrose, urea	Fructose, maltose, magnesium stearate, HCO_3^{-}, PO_4^{3-}, urea, K^{+}, NH_4^{+}, Ca^{2+}, Zn^{2+}, SO_4^{2-}
500	Citrate, lactate	Dextrin, glucose, amylum
50	VB_1	
20		VB_1
5	VB_2	
1	Ascorbic acid	VB_2
0.1	Uric acid	
0.05		Ascorbic acid, uric acid

3.1.5. Effect of the length of reaction coil on MSB analysis

The influence of the reaction coil length was investigated between 5 and 450 cm (1 mm i.d.) at a constant sample flow rate of 2.5 ml min^{-1}. The analytical signal increased with increasing length up to 400 cm, thereafter remaining almost constant. Therefore, a reaction coil that was 400 cm length and 1 mm i.d. was selected for further experiments.

3.1.6. Effect of flow rate

Sample flow-rate was important in the MSB analysis because online conversion of MSB in alkaline medium into sodium bisulfite was necessary. Between 0.5 and 2.5 ml min^{-1}, the analytical signal increased with increasing flow-rate. Therefore, a sample flow-rate of 2.5 ml min^{-1} was recommended. The carrier stream (acidic rhodamine 6G and Tween solutions) flow rate was studied between 1 and 4.5 ml min^{-1}. It was found that the CL intensity increased with increase in flow rate because this CL reaction is a fast process. However, if the flow rate was greater than 3.0 ml min^{-1}, the accuracy became unacceptable. A carrier stream flow-rate of 3.0 ml min^{-1} was recommended.

3.2. Determination of the studied drugs

Under the optimized conditions given above, a series of standard solutions over the concentration range cited in Table 1 was pumped, each as four replicates, to test the linearity of the calibration graph. A plot of the CL intensity versus concentration of studied drugs was linear over the ranges given in Table 1. In order to examine the validity of the method, it was applied to pure samples of studied drugs. The average recoveries ranged from 99.4 ± 1.20 to $101.0 \pm 2.11\%$. R.S.D. ($n = 11$) were 3.4% for 0.05 μg ml^{-1} of MSB, 4.0% for 1 μg ml^{-1} of MSB, 4.7% for 10 μg ml^{-1} of MSB, and 1.5% for 2 μg ml^{-1} of analgin, respectively.

3.3. Study of interferences

The effect of foreign substances was tested by analyzing a standard solution of studied drugs (2 μg ml^{-1}) to which increasing amounts of interfering substances were added. The tolerable concentration ratios with respect to 2 μg ml^{-1} MSB or analgin for interference at 5% level are reported in Table 2. It can be seen that uric acid and ascorbic acid interfere seriously. Therefore, sample pretreatment was necessary when the proposed method was applied to the analysis of the studied drugs in biological samples.

3.4. Analysis of pharmaceutical preparations

3.4.1. Analysis of MSB in injection samples

Due to the reversible equilibrium as shown in Fig. 5, sodium bisulfite was usually added to the commercial injections as stabilizer. Therefore, determination of bisulfite in injections was made firstly. This can be done easily by replacing sam-

Table 3
Results for the determination of MSB in injections

Sample	Amount in sample ($\mu g\ ml^{-1}$)	Standard added ($\mu g\ ml^{-1}$)	Amount found ($\mu g\ ml^{-1}$)	Found labeled (%)	Recovery (%)
1	0.50		0.495	99.0	
	0.50		0.486	97.2	
	0.50		0.492	98.4	
	0.50	0.500	1.004		100.8
	0.50	0.500	1.002		100.4
	0.50	0.500	0.998		99.6
2	1.00		0.932	93.2	
	1.00		0.947	94.7	
	1.00		0.952	95.2	
	1.00	1.000	1.956		95.6
	1.00	1.000	2.001		100.1
	1.00	1.000	1.984		98.4

ple in 0.03 M sodium carbonate by sample in water. The determination and calibration of bisulfite were performed under the following conditions: 0.08 mM Rh6G in 0.06 M H_2SO_4/0.5% Tween/sample or standard solution in water. The true concentration of MSB was calculated in sample, $C_{MSB,\,true}$, using the equation

$$C_{MSB,\,true} = C_{MSB} - AC_1 \quad (1)$$

where C_{MSB} is the sum of MSB concentration and bisulfite concentration expressed as the MSB concentration, C_1 is the concentration of bisulfite, A is converting factor $A = M_{MSB}/M_{HSO_3^-}$, M_{MSB} is the molar weight of MSB, $M_{HSO_3^-}$ is the molar weight of HSO_3^-, $A = 330.3/81 = 4.078$. Recovery standard addition tests and comparison with spectrophotometry [22] were carried out giving the results shown in Tables 3 and 4, respectively. As illustrated in Tables 3 and 4, the proposed method can be satisfactorily applied to the determination of MSB in injection samples.

3.4.2. Determination of analgin in tablets

The proposed method was further applied to the analysis of certain dosage forms containing analgin. The results are shown in Table 5. Recovery tests were carried out on samples to which known amounts of analgin were added. The recoveries for the different concentration levels varied from 96 to 103%, as shown in Table 5.

3.5. Analysis of spiked urine and serum samples

The proposed method allowed the determination of the studied drugs in biological fluids. The Somogyi treatment was carried out with equimolar portions of $Ba(OH)_2$ and $ZnSO_4$ (0.1 M in each case) to remove all reducing substances from serum and urine [23–25]. Using this approach, the recoveries were satisfactory for both drugs. Table 6 shows the results of the recovery studies of studied drugs from spiked urine and serum.

4. Conclusion

The proposed flow system, with oxygen as oxidant, Tween 80 as sensitizer, Rh6G as energy transfer reagent and chemiluminescence detection, was found to be useful for the determination of

Table 4
Results for the determination of MSB in injections[a]

Sample	Proposed method (mg ml^{-1})	Spectrophotometric method (mg ml^{-1})
Injection 1	3.98($\pm$0.7%)	4.00($\pm$1.0%)
Injection 2	3.78($\pm$1.5%)	3.75($\pm$2.8%)
Injection 3	3.80($\pm$3.4%)	3.90($\pm$2.5%)

[a] Values are mean of three replicates ($\pm$R.S.D. %).

Table 5
Results for the determination of analgin in tablets

Sample	Amount (mg)		Added (μg/ml)	Found (μg/ml)	Recovery (%)
	Label (mg)	Found ± S.D. (%) ($n = 5$)			
Tablet 1	500	501 ± 2.5	125	628.5	102.8
			250	746.7	98.7
			500	997.3	99.5
Tablet 2	500	498 ± 1.7	125	620.5	96.4
			250	750.2	100.1
			500	989.9	98.0

Table 6
Determination of the studied drugs in spiked urine and serum

Compound	Conc. Taken (μg/ml)	Found[a] (% ± R.S.D.%)				
		Urine 1	Urine 2	Urine 3	Serum 1	Serum 2
MSB	20	97.2(±2.5)	94.5(±2.8)	96.2(±1.7)		
	30	98.8(±2.3)	93.3(±4.1)	95.3(±0.4)		
	40	97.4(±2.7)	93.2(±3.1)	94.2(±2.0)	92.9(±2.0)	93.9(±1.5)
	60	98.4(±1.0)	94.3(±4.2)	95.7(±1.5)	94.2(±1.5)	94.4(±3.9)
	80				93.6(±3.2)	95.1(±2.3)
Mean ± R.S.D.		97.95 ± 0.76	93.83 ± 0.61	95.35 ± 0.63	93.57 ± 0.47	94.47 ± 0.45
Analgin	10	95.0(±2.1)	90.5(±1.3)	91.4(±0.9)		
	20	94.0(±3.9)	90.9(±2.3)	90.0(±2.5)	94.3(±2.7)	93.8(±0.5)
	30	93.0(±4.4)	88.9(±2.8)	91.6(±1.2)		
	40	93.9(±0.87)	90.2(±0.69)	92.3(±2.0)	94.5(±1.6)	95.6(±3.1)
	60				95.9(±2.4)	95.6(±1.5)
	80				97.4(±3.5)	94.3(±4.0)
Mean ± R.S.D.		93.98 ± 0.56	90.13 ± 0.68	91.33 ± 0.74	95.53 ± 1.18	94.83 ± 0.82

[a] Mean of three replicates.

MSB and analgin. Compared to the use of permanganate or Ce(IV) as oxidant in the conventional CL flow system for pharmaceutical analysis, the present CL flow system is advantageous not only because of its simplicity, but also because it avoids expensive reagent consumption. We have demonstrated that the proposed method can be used successfully for the determination of MSB and analgin in pharmaceuticals and biological fluid.

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