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Simple Spectrophotometric Method for the Quantitative Determination of Uranium

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INSTRUMENTATION, METHODOLOGY,
AND TECHNICAL DEVELOPMENTS

Simple Spectrophotometric Method for the Quantitative Determination of Uranium

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ABSTRACT

A selective, reproducible, and sensitive spectrophotometric method for uranyl determination was developed. The method is based on the selective complexation reaction of UO_2^{2+} ions with 2-(2-benzothiazolylazo)phenol(I), -4-methoxyphenol (II), and -3-hydroxyphenol(III) in the presence of sodium lauryl sulphate. The potentiality of the prepared reagents as new chromogenic ones for the determination of UO_2^{2+} ions are studied by extensive investigations of the optimum conditions favoring the formation of the colored complexes. The method allows the determination of UO_2^{2+} in the range of 0.05–1.60, 0.05–1.40, and 0.05–1.70 $\mu\text{g mL}^{-1}$ at λ_{max} 585, 608, and 599 nm using reagents I, II, and III, respectively. For more accurate results, Ringbom optimum concentration ranges were found to be 0.10–1.60 $\mu\text{g mL}^{-1}$. The molar absorptivity, Sandell sensitivity, detection, and quantification limits are also evaluated. The interfering ions and their tolerance limits have been studied. The method was successfully applied to the determination of uranium from standard ores, geological samples, and synthetic matrices.

Key Words: Uranium determination; Thiazolylazo compounds; Spectrophotometry; Surfactants; Ore analysis.

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INTRODUCTION

In spite of more than 50 years of intense interest in the VI oxidation state of uranium, uranyl UO_2^{2+} , since the commencements of nuclear energy production, there is still a lack of a simple inexpensive method to analyze this element in its most prevalent oxidation state in the environment.

If an available ligand is somewhat equivalent to EDTA for Ca^{2+} and other metal ion analyses, environmental scientists will be able to access to simple ultraviolet-visible equipment to determine UO_2^{2+} in a wide range of samples such as fertilizers, groundwater, biofluids, effluents, etc.

Thus, we sought a relatively inexpensive ligand which was nondangerous and which could be used specifically and accurately for the measurement of UO_2^{2+} in the presence of other metal ions and a range of solvent matrices.

Various organic dyes and reagents have been used for the spectrophotometric determination of UO_2^{2+} in complex materials, such as 4-(2-pyridylazo) resorcinol,^[1] thoronal,^[2] chrome azurol S,^[3] 1-(2-pyridylazo)-2-naphthol,^[4] guaiacol,^[5] 2-(5-bromo-2-pyridylazo)-5-(diethylamino)phenol,^[6] molybdophosphoric acid,^[7] *N-m*-tolyl-*p*-methoxybenzohydroxamic acid,^[8] 4-methyl-4-chlorodibenzoyl-methane,^[9] and 2-hydroxy-1-naphthaldehyde isonicotinoyl hydrazone.^[10] However, most of these methods are tedious, needing rigid control of pH, temperature, reagent, etc., and are also time consuming. These methods have generally low reproducibility and are also not selective, as many of the common ions that are generally found associated with uranium, such as V^{5+} , Zr^{4+} , Fe^{3+} , Cr^{3+} , and SiO_3^{2-} interfere.^[11]

Therefore, in this investigation, an effort was made to develop a simple, rapid, sensitive, reproducible, and selective method for the determination of UO_2^{2+} ions. The method is based on the selective reaction of UO_2^{2+} with three thiazolylazo compounds in the presence of sodium lauryl sulphate. The method was successfully applied to the recovery of uranium from standard ores, geological samples, and synthetic matrices.

EXPERIMENTAL

Apparatus

Absorption spectra were recorded (JASCO 530 V, UV-Visible recording spectrophotometer) using a spectral band-width of 0.5 nm. All measurements were performed by using quartz cells with a path-length of 10 mm. Conductometric measurements were carried out using an YSI model 32 M conductance meter with YSI 3417 dip type cell ($K_{\text{cell}} = 1.0$). pH measurements were made with an Orion research model 601 A/digital ionalyzer pH meter.

Reagents

All chemicals were of analytical-reagent grade. Double distilled water was used throughout.



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Standard solution of uranium(IV) 1×10^{-3} M, was prepared by dissolving an exactly weighed amount of uranyl nitrate in distilled water and was standardized gravimetrically using 8-hydroxyquinoline as precipitating reagent.^[12] A working standard solution ($1.0 \mu\text{g mL}^{-1}$) was prepared from this stock solution by suitable dilution with water.

Sodium lauryl sulphate (SLS) solution, 3.0% w/v, was prepared from SLS obtained from Aldrich Chemical Company.

Thiel buffer solutions of pH's 2.00–12.0 were prepared as recommended previously.^[13]

Reagents I–III solution, 1×10^{-3} M, was prepared by dissolving the appropriate amount of pure reagent in 95% ethanol.

Synthesis of Reagents

Three solutions of 2-amino benzothiazole (7.51 g) were dissolved in 10 mL formic acid and 20 mL of concentrated sulfuric acid, then 30 mL of water were added. To each, a solution of 3.7 g sodium nitrite in 30 mL of water at $0-2^{\circ}\text{C}$ was added dropwise and the mixture was stirred for 1.5 h at $0-2^{\circ}\text{C}$. Separately, phenol (4.71 g) [I], 4-methoxyphenol (6.21 g) [II], and 3-hydroxyphenol (5.51 g) [III] were dissolved in small amounts of methanol and pyridine and cooled to $0-2^{\circ}\text{C}$. The solution was added dropwise to one of the above diazotized solution with vigorous stirring. The mixture was stirred for an additional 2.0 h in an ice-bath and allowed to stand overnight. The precipitate formed was filtered off and first purified by base-acid recrystallization method and further purified by recrystallization from dimethylformamide (DMF)-water (1:1). A red solid, which decomposes at 234, 217, and 247°C for I, II, and III, respectively, were obtained in a yield of about 40–43%. Their structures were confirmed by IR and $^1\text{H-NMR}$ spectrometry.

General Procedure

An aliquot of the solution containing up to $17 \mu\text{g}$ of UO_2^{2+} is placed in a 10 mL calibrated flask, 4.0 mL of thiel buffer solution of pH 6.5, 8.0, and 6.0 for reagent I, II, and III, respectively. 2.0 mL of 3.0% SLS solution and 2.5 mL of 1×10^{-3} M reagent solution were added and the mixture diluted to 10 mL with bidistilled water, under mixing. After 2.0 min, the absorbance at 585, 608, and 599 nm for I, II, and III complex was measured in 10 mm cell against a reagent blank prepared in a similar way without metal ions.

Procedure for Standard Ores, Geological Samples, and Synthetic Matrices

The standard ore sample was prepared according to the literature.^[14] Accurately weighed amounts of the geological samples were taken and digested with HF and concentrated HCl. The excess of F^{-} ions was removed by nitric acid treatment as



mentioned above. After drying, the sample was dissolved in distilled water and diluted to a fixed volume. An aliquot of the sample solution was shaken with ethylacetate in the presence of aluminium nitrate to separate uranium from other metal ions.^[15] The metal was stripped from the organic phase by shaking with distilled water. The metal content was determined in the sample by the recommended general procedure.

RESULTS AND DISCUSSION

Absorption Spectra

The uranyl-reagent (I–III) complex has a low solubility in water but when the reaction occurs in presence of surfactant SLS, it forms a highly soluble pink complex with maximum absorbance at 585, 608, and 599 nm, in the pH range 5.5–8.5. Hence, all measurements were made at pH 6.5, 8.0, and 6.0 using reagent I, II, and III, respectively, since the absorbance is high and more stable (Fig. 1). The complexes are instantaneously formed, solubilized and stable for at least one day. The absorption spectra of the reagents and their UO_2^{2+} complexes in the presence of SLS indicated that the band in the visible region of the free reagent is bathochromically shifted in the presence of UO_2^{2+} ions.

Investigations were carried out to establish the most favorable conditions to achieve maximum color development in the quantitative determination of UO_2^{2+} ions. The influence of each of the following variables on the reaction was tested.

Effect of pH

The experimental results demonstrated that the absorbance of UO_2^{2+} complex is maximum and constant in the pH range 6.0–7.0, 7.5–8.5, and 5.5–6.5 and a

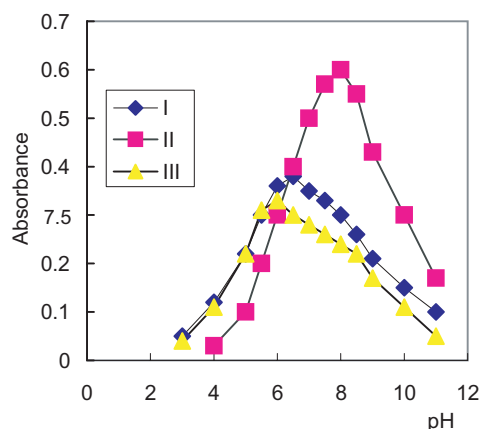


Figure 1. Effect of pH on the complexation reaction of 2.5×10^{-4} M reagents I, II, and/or III with $1.0 \mu\text{g mL}^{-1}$ of UO_2^{2+} .



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pH 6.5, 8.0, and 6.0 were adopted for complexes of reagents I, II, and III, respectively. Addition of 2.0–6.0 mL of thiel buffer per 10 mL solution did not affect the absorbance and the use of 4.0 mL of buffer was adopted.

Effect of Surfactants

Both in the absence of surfactants as well as in the presence of the cationic surfactants, cetylpyridinium chloride (CPC), cetyltrimethyl ammonium bromide (CTMAB), the UO_2^{2+} -reagent chromogenic system shows low absorption, whereas in the presence of nonionic surfactants, the absorption of the chromogenic system increases markedly. Various nonionic and anionic surfactants enhance the absorbance in the following sequence: sodium lauryl sulphate > sodium dodecyl sulfate (SDS) > sodium dodecylbenzene sulfonate (SDBS) > polyethylene glycol phenylether (OP) > Triton X-100 > Tween-80.

Addition of appropriate amounts of various organic solvents did not affect the absorbance of the complex formed. Hence, SLS is the best sensitizing agent. Moreover, 1.5–2.5 mL of 3.0% SLS solution giving a constant and maximum absorbance, hence the use of 2.0 mL was selected (Fig. 2).

Effect of Reagent Concentration

The absorbance of a series of solutions containing $1.0 \mu\text{g mL}^{-1} \text{UO}_2^{2+}$ and various amounts of 10^{-3} M reagent solution were measured. It was found that 2.0 mL of 10^{-3} M reagent is sufficient to complex $1.0 \mu\text{g mL}^{-1} \text{UO}_2^{2+}$. With 2.5 mL reagent concentration, the absorbance was essentially constant. Therefore, 2.5 mL was used since the results are highly concordant at this level. The time for the absorbance to reach a stable value was 2.0 min, after mixing at room temperature and the absorbance was stable for at least one day.

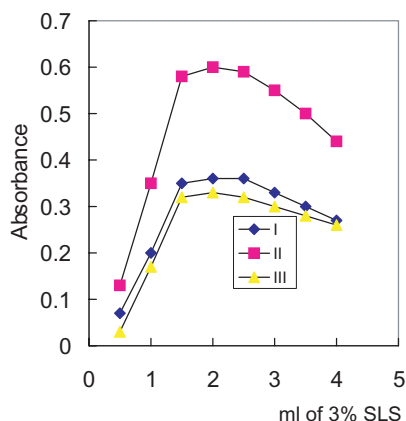


Figure 2. Effect of SLS concentration on complexes formed between $2.5 \times 10^{-4} \text{ M}$ reagents I, II, and/or III with $1.0 \mu\text{g mL}^{-1}$ of UO_2^{2+} .



Sequence of Additions

The most favorable sequence is “ UO_2^{2+} -buffer-SLS-reagent” to obtain the highest color intensity and least time for developing maximum absorbance. All other sequences needed longer times and gave lower absorbance. The complexes with this sequence remain stable for at least one day, whereas using other sequences, the complexes remain stable for 4.0 h only.

Composition of the Complexes

Under the optimum conditions of pH, reagent and surfactant concentration, and sequence of addition described above, the UO_2^{2+} -reagent ratio was found to be 1:2 (M: R), using the molar ratio and continuous variation methods. These results were further confirmed by the data obtained from conductometric titration. The conditional formation constants ($\log K$), calculated from Harvey and Manning equation^[16] applying to the data obtained from the molar ratio and continuous variation methods, were found to be 6.85, 8.20, and 5.90 for I, II, and III- UO_2^{2+} complexes, respectively. These values indicated that the stability of the complexes increases in the order $\text{II} > \text{I} > \text{III}$. A representative structure for the complex formed taking I as an example is shown below.

Characterization of the Complexes

All the complexes are intensity colored air stable. The IR spectrum of the azo derivatives shows a broad band at $3400\text{--}3500\text{ cm}^{-1}$ due to the stretching frequency of the phenolic OH group, while additional absorption bands at 1410 and 1240 cm^{-1} is due to the coupled $\nu_{\text{C-O}}$, δ_{OH} in plane frequency.

The strong band at $1450\text{--}1550\text{ cm}^{-1}$ is due to $\nu_{\text{N}=\text{N}}$. The IR spectra of the complexes show the presence of some new bands and shifts of the characteristic modes. The IR spectrum of azo-uranyl complexes shows a change in the position of azo $\text{N}=\text{N}$ group indicating the involvement in coordination, the new bands at 910 and 820 cm^{-1} are assigned to the asymmetric and symmetric stretching vibration of uranyl entity. In the electronic spectrum of the azo-uranyl complex, there are overlapped by the fairly strong ligand (azo dyes) to metal charge transfer band. This charge transfer transition probably occurs from the π -orbital of the azo to the f -orbitals of uranium. Elemental analysis and IR spectra reveals that azo-dye react with uranyl to forms the following structural complex (see Sch. 1).

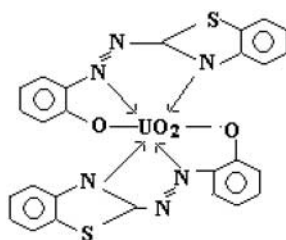
Analytical Data

The calibration graph was constructed following the general procedure. The linear regression equation was applied and the values of slope, intercept, and correlation coefficient obtained are recorded in Table 1. Beer's law was obeyed in the range recorded in Table 1 for each system. The calculated apparent molar



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Scheme 1.

Table 1. Spectral characteristics for UO_2^{2+} -reagents (I–III) complexes.

Parameter	Reagent		
	I	II	III
pH	6.5	8.0	6.0
λ_{max} (nm)	585	608	599
Stability (h)	24	36	30
Concentration (range/ $\mu\text{g mL}^{-1}$)	0.05–1.60	0.05–1.40	0.05–1.70
Ringbom conc. (range/ $\mu\text{g mL}^{-1}$)	0.10–1.50	0.15–1.25	0.10–1.60
Detection limits (ng mL^{-1})	16	14	18
Quantification limits (ng mL^{-1})	50	47	53
Molar absorptivity ($\text{L mol}^{-1} \text{cm}^{-1}$)	1.03×10^5	1.62×10^5	8.74×10^4
Sandell sensitivity (ng cm^{-2})	2.62	1.67	3.09
Regression equation ^a			
Slope	0.38	0.60	0.33
Intercept	–0.007	0.011	0.009
Correlation coefficient	0.9994	0.9996	0.9990
Recovery (%)	99.10 ± 1.50	99.70 ± 1.00	100.20 ± 1.20
Relative standard deviation ^b (%)	1.24	0.92	1.47
Calculated <i>t</i> -value (2.45) ^c	1.63	1.09	1.86
Calculated <i>F</i> -value (4.39) ^c	3.25	2.66	3.59

^a $A = a + bC$, where *C* of the concentration in $\mu\text{g mL}^{-1}$.^bAverage of eight determinations.^cTheoretical values for 95% confidence limits.

absorptivity and Sandell sensitivity for each uranyl complex with reagents (I–III) are included in Table 1. For more accurate analysis, optimum concentration ranges were calculated using Ringbom method and recorded in Table 1. The relative standard deviation at a concentration level $1.0 \mu\text{g UO}_2^{2+}$ (eight repeated determinations) was calculated and listed in Table 1, and found in the range 0.92–1.47%.

The standard deviation of the absorbance measurements was calculated from a series of 13 blank solution. The limits of detection ($K=3$) and of quantification ($K=10$) of the method were established according to the IUPAC definitions



($C_1 = KS_0/s$, where C_1 is the limit of detection, S_0 is the standard error of blank determination, s is the slope of the standard curve and K is the constant related to the confidence interval)^[17] and the values were recorded in Table 1.

The performance of the proposed methods was assessed by calculation of t - and F -values compared with that using Arsenazo III^[18] for 95% confidence limits with seven degrees of freedom.^[19] The results showed that there was no significant difference between the proposed and the Arsenazo III methods.

Selectivity

The previously reported spectrophotometric methods suffer from interference from many metal ions. These methods require prior separation and are tedious and time consuming. The conventional thoronal method^[2] suffers from interference from Fe, Cr, V, Zr, Cu, Hg, Mn, Ce, Th, and Mo, etc. In this investigation none of the above except Th^{4+} interfere; the interference can be masked effectively with 0.2 mL of 10% tartaric acid solution. Using reagents (I–III) as a complexing reagent, at least a 50-fold molar excess of the above mentioned elements can be tolerated in real samples.

Effect of Ions

The effect of diverse ions on the determination of $1.0 \mu\text{g mL}^{-1}$ by the proposed procedure were studied. A large number of ions do not interfere. Th^{4+} interferes but the interference can be overcome as described above. Fluoride when present above five fold excess of UO_2^{2+} prevents the formation of the complex. Therefore, F^- when present in excess, is removed by nitric acid treatment, as recommended previously.^[20] The tolerance limits of the various diverse ions (in $\mu\text{g mL}^{-1}$) causing an error less than $\pm 3.0\%$ are given in Table 2.

Table 2. Tolerance limits of various ions on the determination of uranium.

Ion added	Tolerance limits ^a ($\mu\text{g mL}^{-1}$)	Ion added	Tolerance limits ^a ($\mu\text{g mL}^{-1}$)
Cl^- , CH_3COO^-	20,000	Na^+ , Li^+ , K^+	25,000
PO_4^{3-} , SO_4^{2-}	18,000	Ca^{2+} , Mg^{2+}	20,000
Tartrate	15,000	Mn^{2+} , Pb^{2+}	17,000
NO_3^- , ClO_4^-	12,000	Cd^{2+} , Sn^{2+}	15,000
Benzoate	9,000	Ga^{3+} , Al^{3+}	12,000
Citrate	7,500	Fe^{3+} , Cr^{3+}	8,000
Oxalate	6,000	V^{5+} , Zr^{4+}	6,500
Borate, $\text{S}_2\text{O}_3^{2-}$	5,000	Ce^{4+} , Mo^{6+}	5,000
Succinate, urea	2,500	Cu^{2+} , Co^{2+}	3,000
Thiourea	2,000	Ni^{2+} , Pd^{2+}	1,500
Ascorbic acid	1,300	La^{3+} , Sc^{3+}	1,000
F^-	100	Th^{4+} ^b	400

^aCausing an error of less than $\pm 3.0\%$.

^bMasked with tartaric acid.



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Table 3. Determination of uranium in standard ores, geological, and synthetic matrices.

Sample	Uranium found ^a ($\mu\text{g mL}^{-1}$)				Certified value ($\mu\text{g mL}^{-1}$)
	I	II	III	Arsenazo III	
USGS G-2 ^a	1.97	2.02	2.01	1.95	2.0
	$t^c = 1.73$	$t^c = 1.21$	$t^c = 1.88$		
	$F^c = 3.25$	$F^c = 2.83$	$F^c = 3.90$		
Granite rock ^b	1.99	1.98	1.96	2.07	2.0
	$t^c = 1.56$	$t^c = 1.09$	$t^c = 1.81$		
	$F^c = 3.11$	$F^c = 2.34$	$F^c = 3.60$		
Monazite sand ^b	0.33%	0.32%	0.34%	0.30%	0.33%
	$t^c = 1.74$	$t^c = 1.36$	$t^c = 1.58$		
	$F^c = 3.46$	$F^c = 3.05$	$F^c = 3.33$		

^aUnited States Geological Survey Standard.^bGeology Department, Faculty of Science, Benha University, Egypt.^cTheoretical values for t - and F -values for 95% confidence level and five degrees of freedom are 2.57 and 5.05.

Analytical Applications

The proposed method was applied to the recovery of uranium from standard ores, geological samples, and synthetic matrices. The uranium content was determined in the sample by the recommended procedure and the results were compared with those obtained using Arsenazo III.^[18] The recovery of UO_2^{2+} from synthetic matrices, prepared similarly to enriched ores, was examined and was found to be satisfactory. The results obtained are summarized in Tables 3 and 4.

The results obtained by the proposed method were compared with those using Arsenazo III^[18] by t -test for accuracy and the F -test for assessment of precision^[19] for five degrees of freedom and 95% confidence level. The calculated values did not exceed the corresponding theoretical values, indicating insignificant differences between the results.

CONCLUSION

It is clear that reagent II is highly superior compared to the other examined reagents being highly sensitive ($1.62 \times 10^5 \text{ L mol}^{-1} \text{ cm}^{-1}$), exhibiting longer stability of color (36 h) and lower relative standard deviation % (0.92). A comparison of the possibilities of the reagents (I–III) under consideration for the spectrophotometric determination of UO_2^{2+} with those of the other methods (Table 5) shows that, the proposed reagent (II) is one of the most sensitive reagents for UO_2^{2+} determination. Most foreign ions do not interfere and the selectivity of the method is good in the

**Table 4.** Recovery of uranium from synthetic matrices.

Solution number	Ions present	Found ^a			Arsenazo III
		I	II	III	
1	U ⁶⁺ 10 µg + Zr ₄ ⁺ 500 µg + Cr ³⁺	10.05	9.96	10.10	9.82
	200 µg + Al ³⁺ 1.0 µg + Mo ⁶⁺	<i>t</i> ^b = 1.70	<i>t</i> ^b = 1.22	<i>t</i> ^b = 1.22	
	2000 µg + Co ²⁺ 500 µg	<i>F</i> ^b = 3.56	<i>F</i> ^b = 2.85	<i>F</i> ^b = 3.14	
2	U ⁶⁺ 2 µg + Zr ₄ ⁺ 100 µg + Cr ³⁺ 40 µg +	2.01	2.00	1.99	2.05
	Al ³⁺ 170 µg + Mo ⁶⁺ 400 µg +	<i>t</i> ^b = 1.25	<i>t</i> ^b = 0.85	<i>t</i> ^b = 1.08	
	Co ²⁺ 135 µg	<i>F</i> ^b = 2.89	<i>F</i> ^b = 2.27	<i>F</i> ^b = 2.51	

^aAverage of six determinations.^bTheoretical values for *t*- and *F*-values for 95% confidence level and five degrees of freedom are 2.57 and 5.05.**Table 5.** Comparison of reagents for spectrophotometric determination of uranium.

Reagent	λ_{\max}	ϵ , L mol ⁻¹ cm ⁻¹	Ref.
<i>N</i> -hydroxy, <i>N,N'</i> -diphenylbenzamidine	490	8.80×10^4	[21]
4-(2-thiazolylazo)-6-chlororesorcinol	553	3.00×10^4	[22]
Di-2-pyridyl ketone nictinoyl hydrazone	373	2.13×10^4	[23]
Di-2-pyridyl ketone thiophenoyl hydrazone	389	2.33×10^4	[23]
Arsenazo III	650	5.00×10^4	[2]
Reagent I	585	1.03×10^5	This work
Reagent II	608	1.62×10^5	This work
Reagent III	599	1.67×10^5	This work

presence of tartaric acid. The proposed method was successfully applied to the determination of UO₂²⁺ in standard ores, geological samples, and synthetic matrices and compared with Arsenazo III^[18] method.

ABBREVIATIONS

CPC	Cetylpyridinium chloride
CTMAB	Cetyltrimethyl ammonium bromide
DMF	Methylformamide
IR	Infrared
IUPAC	International Union for Pure and Applied Chemistry
NMR	Nuclear magnetic resonance
SDS	Sodium dodecyl sulfate
SDBS	Sodium dodecylbenzene sulfonate
SLS	Sodium lauryl sulfate



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