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COCRYSTALLIZATION OF ULTRAMICRO QUANTITIES OF
ELEMENTS WITH 2-MERCAPTOBENZIMIDAZOLE

DETERMINATION OF GOLD IN SEA WATER

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ADMINISTRATIVE INFORMATION

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ABSTRACT

The cocrystallization of ultramicro quantities of 25 diverse elements with 2-mercaptobenzimidazole was investigated with the aid of radio-tracers. Under optimum conditions Sn, Hg, Ag, Ta and Au were recovered in high yield.

Distribution coefficients between mother liquor and solid phase were calculated for gold and were found to vary inversely with the degree of crystallization and the hydrogen ion concentration.

The cocrystallization process was applied to the isolation and subsequent determination of the natural gold abundance in sea water. After radiometric correction for chemical yield, the concentration of gold was calculated to be $0.068 \pm 0.003 \mu\text{g/l}$.

SUMMARY

Determination of trace element abundances in sea water requires the concentration and ultimate isolation of an element from large quantities of salts and sample volumes. The cocrystallization of minute quantities of elements with certain organic reagents offers an effective means of achieving the separation.

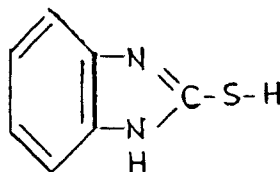
With the use of radioactive tracers, a method was developed for the high-yield recovery of tin, mercury, silver, tantalum and gold from very dilute solution by cocrystallization with 2-mercaptobenzimidazole. The method was used as a preliminary separation step in the determination of the concentration of gold in sea water.

INTRODUCTION

In the detection, control or study of the radioactive contamination in a marine environment, sensitive analytical methods are required. This requirement follows from the magnitude of the dilution factors involved. To attain the requisite sensitivity, a preliminary concentration step generally is incorporated in the analytical scheme.

In this laboratory the concentration of ultramicro quantities of elements from solution by cocrystallization with organic agents has been investigated.^{1a-f} The process has been applied to the collection of radioelements as well as the characterization of the trace element abundance in seawater.

Cocrystallization of various elements with 2-mercaptobenzimidazole, whose structure is shown below, is described in this report. This reagent has limited solubility, is readily crystallized from aqueous solution, and forms highly insoluble complexes with certain metals. These properties usually favor effective cocrystallization.



The influence of pH upon the cocrystallization of 26 diverse elements was studied. To permit the quantitative measurement of trace quantities of these elements, radioactive tracers were used. The distribution of gold between the mother liquor and solid phase was determined by the crystallization of different quantities of carrier from solution. Finally, the system was applied in a determination of the natural abundance of gold in seawater.

EXPERIMENTAL

Reagents

2-Mercaptobenzimidazole Solution: A weighed quantity of 2-mercaptobenzimidazole (K & K Laboratories, Jamaica, N.Y.) was dissolved in absolute ethyl alcohol at a concentration ranging from 0.3 to 1.5 %.

Solutions of Different Hydrogen Ion Concentration: Solutions of pH 1 through 3 consisted of hydrochloric acid of the appropriate normality. Solutions of pH 4 and 5 were mixtures of acetic acid (AR) and sodium acetate (AR) each about 0.1 M.

Tracers

The following radioactive tracers were furnished by Oak Ridge National Laboratory:

In HCl solution: $Ce^{144}(\text{III})$, $Sc^{46}(\text{III})$, $In^{114}(\text{III})$, $Y^{91}(\text{III})$, $Sn^{113}(\text{II})$, $Sb^{124}(\text{III})$, $Fe^{59}(\text{III})$, $Zn^{65}(\text{II})$, $Cr^{51}(\text{III})$, $Cs^{137}(\text{I})$, $Ir^{192}(\text{IV})$, $Co^{60}(\text{II})$, $Hf^{181}(\text{IV})$, $Se^{75}(\text{IV})$, $Ru^{106}(\text{III})$.

In oxalic acid: $Zr^{95}(\text{IV})$. (This radioelement was separated from Nb^{95} just before use (2)).

In nitric acid: $Sr^{85}(\text{II})$, $Ag^{110}(\text{I})$, $Tl^{204}(\text{I})$, $Hg^{203}(\text{II})$.

In KOH: $Ta^{182}(\text{V})$, $W^{185}(\text{VI})$.

Supplied by Nuclear Science and Engineering Corporation were $Mn^{54}(\text{II})$ and $Au^{195}(\text{III})$ each in HCl.

Prepared in the laboratory by the reaction $U^{236}(n,\gamma) U^{237}$ was $U^{237}(\text{VI})$ in nitric acid.

The purity of the gamma-ray emitters was established by gamma-ray pulse-height analysis. Some of the tracers contained inert carrier. In such cases the quantity of tracer introduced into the test solution was limited so that the final concentration of the element was 10^{-8} g/ml or less.

Method

The influence of hydrogen ion concentration upon the cocrystallization of various radioclements with 2-mercaptobenzimidazole was studied

at pH 1, 3 and 5. (More alkaline media were not examined since the reagent becomes increasingly soluble as the pH is raised). To 11 ml of the solution of definite pH containing the inorganic microcomponent was added 1 ml of a 1.5 % solution of 2-mercaptobenzimidazole. The solution was cooled at 5°C for 48 hours. The crystals which formed were collected by filtration and dissolved in ethyl alcohol. The recovery of the radioelement in the solid phase was determined by measurement of the gamma-ray activity of the alcoholic solution in a well scintillation counter and by comparison of the count with a radioactivity control of the same volume. In a number of cases this solution was also submitted to spectrophotometric analysis to determine the fraction of organic reagent which crystallized.

The distribution of gold between solid phase and mother liquor was studied at different hydrogen ion concentrations by crystallizing different fractions of the organic carrier from solution. The procedure was the same as described above except that, to effect variable yields of crystallized carrier, the concentration of 2-mercaptobenzimidazole in alcoholic solution added to a sample varied from 0.3-1.5 %.

Spectrophotometric Determination of 2-Mercaptobenzimidazole

The distribution of 2-mercaptobenzimidazole between solid phase and mother liquor was determined photometrically. With a Beckman spectrophotometer the maximum spectral absorption occurred at 327 m μ when the reagent was dissolved in absolute ethyl alcohol. The absorbancy at this wavelength of 0.98, 1.95, 3.90 and 5.85 ppm was 0.151, 0.300, 0.584 and 0.905, respectively with adherence to Beers Law.

RESULTS AND DISCUSSION

The recovery of the tracer quantities of elements from solutions of different hydrogen ion concentration with crystallized 2-mercaptobenzimidazole is shown in Table I. (Under the conditions of crystallization, 97 % of the reagent was recovered at pH 1 and 3, and 93 % at pH 5.) Tin (pH 5), Hg (pH 1 through 5), Ag (pH 1 through 5), Ta (pH 1 through 3) and Au (pH 1) were recovered with high yield. With several exceptions the other elements were carried only minimally. However, it is clear that the cocrystallization process is useful primarily as a mechanism of concentration rather than a method for purification.

TABLE 1

The Recovery of Various Elements With 2-Mercaptobenzimidazole Upon Crystallization From Solutions of Different pH

Element	Recovery (%)		
	pH 1	pH 3	pH 5
Cs	0.6	5.9	0.8
Sr	3.4	1.5	1.6
Se	29.8	14.6	16.2
Sb	2.2	6.9	4.9
Sn	92.3	91.2	99.8
In	4.8	8.6	6.6
Tl	5.1	7.3	17.5
Zn	2.9	3.1	2.0
Hg	98.7	98.8	98.7
Ag	99.0	99.0	99.5
Co	0.6	1.7	2.0
Ir	3.1	1.4	3.7
Fe	2.8	3.7	7.0
Ru	29.1	35.1	41.7
Mn	2.5	0.9	2.0
Cr	3.5	5.8	5.9
W	1.1	4.3	3.5
Ta	97.8	98.1	95.4
Zr	1.3	4.8	28.7
Hf	66.8	71.8	80.2
Au	99.4	93.5	81.5
Sc	5.5	6.5	7.3
Y	3.5	6.8	3.7
Ce	2.7	3.0	3.1
U	14.9	13.7	13.8

The relation of the logarithmic (λ) and homogeneous (D) distribution coefficients^{3,4} for gold to the carrier recovery and hydrogen ion concentration is shown in Table II. The coefficients were calculated according to the equations:

$$\log \frac{M_i}{M_f} = \lambda \log \frac{C_i}{C_f}$$
$$\frac{M_i - M_f}{M_f} = D \frac{C_i - C_f}{C_f}$$

where M and C represent the microcomponent and carrier, i and f initial and final quantities in solution. At a constant hydrogen-ion concentration the distribution coefficient, as calculated by either equation, is in general inversely related to the degree of crystallization. The **inconstancy** of the homogeneous distribution coefficient indicates that redistribution of the microcomponent by diffusion and recrystallization was at best, incomplete. Furthermore the relationship between the distribution coefficients and the degree of crystallization, as was previously observed,^{1e} is probably attributable to a disproportionate concentration of gold in the solid phase during the nucleation process, compared with that accumulated during crystal growth after supersaturation was relieved. Also, for any degree of crystallization the distribution coefficient is inversely related to the pH. To account for this observation, solubility relationships between the organic reagent and its gold chelate were determined under the experimental conditions.

The reagent and the gold compound of known specific radioactivity each was stirred separately for 8 hours at 5°C in solutions of specified pH containing 8.3 % ethyl alcohol (v/v). The solubility of the gold compound was determined by radiometrically measuring the inorganic component in the saturated solution, while the solubility of 2-mercapto-benzimidazole was estimated spectrophotometrically. Over the range of hydrogen ion concentrations studied the solubility of the organic compound increased slightly as the pH was raised (Table III). While the metallic compound showed the same relationship, the effect was markedly more pronounced. According to Fajans Rule⁵ the differential effect of pH upon the respective solubilities would manifest itself by greater enrichment in more acid systems, and in fact precisely this result was observed.

TABLE III

Logarithmic (λ) and Homogeneous (D) Distribution Coefficients for the
Cocrystallization of Gold with 2-Mercaptosuccinimide from Solutions
of Different pH

Crystal Recovered (%)	Distribution Coefficients							
	pH 1		pH 2		pH 4		pH 5	
	λ	D	λ	D	λ	D	λ	D
2.3	22.1	23.3						
5.0	14.2	20.1						
18.2	7.2	10.0						
48.5			2.7	3.4				
50.0								
54.0	3.4	10.0						
62.8	3.3	15.3						
66.8			1.0	1.1				
68.9								
73.4	2.0	12.3						
75.0	2.5	14.7						
77.7			1.7	1.5				
78.1								
81.0			1.3	2.4				
81.5								
94.1								

TABLE IV

Distribution of 2-Mercaptosuccinimide in the
Cocrystallization of Gold from Solutions of Different pH

pH	Succinimide (Mg/g Au)	
	Crystal	Solution
1.0	2.05 × 10 ⁻⁴	1.0 × 10 ⁻⁴
2.0	2.32 × 10 ⁻⁴	1.0 × 10 ⁻⁴
4.0	2.5 × 10 ⁻⁴	1.0 × 10 ⁻⁴
5.0	2.7 × 10 ⁻⁴	1.0 × 10 ⁻⁴

DETERMINATION OF GOLD IN SEAWATER

The abundance of gold in seawater has been extensively studied since the work of Sonstadt in 1872.⁶ Most of the analytical procedures depend upon the separation of gold from the salt medium by coprecipitation with lead or iron compounds. A radioactivation method recently described⁷ does not depend upon a preliminary separation. However the variability of results in all these procedures indicates a requirement for improvement.

The cocrystallization of gold with 2-mercaptobenzimidazole was applied to the development of a reliable analytical method. A preliminary experiment was performed in seawater containing Au¹⁹⁵ tracer to determine the quantity of reagent required for effective cocrystallization.

Different volumes of ethyl alcohol, which contained the organic reagent at a concentration of 50 mg/ml, were added to 200 ml of seawater adjusted to pH 1 with hydrochloric acid. The sample was maintained at 5° C for 48 hours. Crystals which formed were collected on sintered glass and solubilized with nitric acid. The recovery of gold was determined by comparing the gamma-ray count of this sample with a radioactive control diluted to the same volume. Gold cocrystallized to the extent of 65.6, 97.2, 98.8 and 99.0 % when 0.6, 0.8, 1.0 and 1.6 ml of organic reagent were used, respectively.

Procedure

Surface seawater was collected in 20-liter polyethylene bottles 40 miles due west of San Francisco. Each bottle was acidified with 200 ml of conc. hydrochloric acid to pH 1 at the time of collection. (Studies with tracer show that under such acid conditions gold is not adsorbed by the walls of the container). To correct for chemical yield a measured quantity of Au¹⁹⁵ was added 16 hours after collection and the bottles were stored for 3 weeks. Then 5 g of 2-mercaptobenzimidazole

in 100 ml of ethyl alcohol were added slowly to each of six 20-liter containers; the bottles were placed in a cold room (0 to 5°C) for two days. Crystals were collected by filtration, combined, and dissolved with 300 ml of conc. nitric acid.

Organic matter was removed by acid oxidation steps as follows: The nitric acid solution was boiled gently for several hours. After partial oxidation the sample was cooled in an ice-bath and the insoluble degradation products were removed by filtration through sintered glass. To the clear solution were added 100 ml of conc. nitric acid and 20 ml of conc. sulfuric acid and the solution was boiled vigorously until SO₃ fumes evolved and it appeared colorless.

The completely oxidized solution was separated into three unequal portions and each was treated individually as follows: To prepare for purification of gold by solvent extraction, 200 ml of distilled water and 50 ml of conc. hydrochloric acid were added to the sulfuric acid solution. The solution was shaken twice with 200 ml portions of ethyl ether. The ether extracts were combined and evaporated to dryness at room temperature.

Further purification was effected by coprecipitating the gold with tellurium. The residue, after ether evaporation, was dissolved in 10 ml conc. hydrochloric acid and 40 ml of distilled water were added. Tellurium tetrachloride (0.2 mg) was introduced and the tellurium metal was precipitated by treatment with stannous chloride according to the method of Sandell.⁸ The tellurium precipitate was dissolved in 2-3 ml of hot aqua regia and the solution was evaporated to near-dryness. Aqua regia (0.5 ml) was again added and then completely evaporated in vacuo in a desiccator at room temperature.

A colorimetric method was adapted⁹ to the quantitative assay of gold in the sample aliquot. To the residue were added 2.5 ml of water, 1 ml of a solution consisting of 9 % sodium bisulfite (anhydrous) and 20 % sodium sulfite (anhydrous), 0.25 ml of 1 % sodium fluoride solution, 0.3 g sucrose, 0.30 ml of acetone, and 0.250 ml of p-dimethylaminobenzalrhodanine (9 mg/100 ml ethyl alcohol). The solution was diluted to 5 ml with distilled water in a volumetric flask. One ml of this solution was removed to determine radiometrically the quantity of seawater represented by the sample aliquot. The absorbancy of the remaining solution was measured in a Beckman spectrophotometer at 470 mμ. With the use of the absorbancy values 0.023, 0.053 and 0.109 for gold solutions at concentrations of 0.23, 0.45 and 0.90 ppm, respectively, the concentration of gold in the sample was determined.

Results

The results of the determinations of the three aliquots are shown in Table IV. The average concentration of gold was 0.068 ± 0.003 $\mu\text{g}/\text{l}$, a value within the normal range for seawater.¹⁰ A blank analysis was performed on all the materials that entered the analysis for a sample volume of 25 liters. The assay indicated that the quantity of gold introduced by way of reagents was within the indicated experimental error of the gold determinations; therefore, its effect upon the seawater determinations was disregarded.

TABLE IV

The Gold Content of Seawater

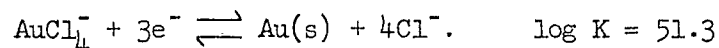
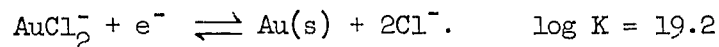
Seawater Sample Volume (liters)	Gold Content ($\mu\text{g}/\text{liter}$)
23.1	0.069
30.6	0.065
36.1	<u>0.070</u>
	Avg. 0.068 ± 0.003

Discussion

With regard to the reliability of these measurements, palladium and platinum metals which interfere with color development in the quantitative method are completely eliminated by ether extraction.¹¹ Other interfering elements such as copper, iron and lead are removed from the system in the tellurium precipitation step. Moreover spectrographic analysis of the combined aliquots after the colorimetric determination revealed besides gold, detectable quantities of magnesium only as an extraneous contaminant. This element is without influence in the colorimetric determination of gold.

Another consideration of importance involves the radiometrically determined chemical yield correction. The validity of this correction presumes that the natural gold in seawater and the radioactive tracer

introduced are chemically identical. The probable chemical form of gold in the acidified seawater sample was determined from the pertinent equilibrium constants (20°C, zero activity).¹²



By substitution of the appropriate values for the pH adjusted seawater: pE = 19.6*, and $\log [\text{Cl}^-] = \log [0.55 \text{ (natural to seawater)} + 0.1 \text{ (from hydrochloric acid added)}] = -0.19$, and neglecting the activity coefficients, the $\log [\text{AuCl}_2^-] = -0.2$ and $\log [\text{AuCl}_4^-] = 6.7$. Clearly AuCl_4^- predominates at equilibrium. The radiometric correction appears valid since the radioactivity was introduced in this chemical form.

The separation of gold from seawater by cocrystallization with 2-mercaptobenzimidazole provides several important advantages over the usual coprecipitation methods with inorganic carriers. The crystallization is performed at pH 1, a condition which completely obviates the difficulties which arise from wall-adsorption of gold at higher pH. Further, more than 95 % of the gold is carried by the crystalline phase. The method of crystallization is simple and the crystals are easily separated from the mother liquor. Since the major constituents in seawater are not cocrystallizable, this first step in the process eliminates the large bulk of salts. Finally the ability to remove the organic carrier by acid oxidation facilitates the ultimate isolation.

*pE = $-\log e^-$

As described by Sillen¹² in seawater the pE is determined by the equilibrium with atmospheric oxygen and is 19.6 for seawater adjusted to pH 1.

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