NASA-CR-193481

DETERMINATION OF TRACE ELEMENTS IN TRIGLYCINE SULFATE SOLUTIONS

GRANT NO. NAG08-204

TECHNICAL FINAL REPORT

SUBMITTED TO

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MARSHALL SPACE FLIGHT CENTER, ALABAMA 35762**

SUBMITTED BY

SHAWKY H. TADROS

July 31, 1993

Shawky H. Tadros, Ph.D.
Project Manager

Jeanette Jones, Ph.D.
VP, Reseach and Development

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Acknowledgement

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My student Séan Hanley also served as a competant, dependable assistant.

A B S T R A C T

Ten elements were divided into 2 groups. The elements in the first group included iron, nickel, Chromium, manganese, copper, and gold,. The elements in the second group included zinc, cobalt, lead, Cadmium, and gold.

Five ppm of each element in each group was spiked in a 1% triglycine sulfate (TGS)solution. Glycine was removed with 1-naphthyl isocyanate in ether medium.The glycine derivative 1-naphthyl isocyanate glycine was removed by filtration, and the filtrates were analyzed for the different elements.

Analysis of these elements was performed by using the 5100 Pekin-Elmer Atomic Absorption Spectrophotometer.

The result of these experiments was the observation that there was a decrease in the concentration of chromium and gold, which was interpreted to be due to the chelation of these elements by the derivative 1-naphthyl isocyanate glycine.

Further research is needed to determine the concentration of other elements in triglycine sulfate (TGS) solutions. These elements will include lithium, sodium, rubidium, magnesium, calcium, strontium, barium, aluminum, and silicon.
These are the most likely elements to be found in the sulfuric acid used in manufacturing the TGS crystal.

Moreover, we will extend our research to investigāte the structural formula of the violet colored chelated compounds, which had been formed by interaction of the derivative 1-naphthyl isocyanate glycine with the different elements, such as gold, chromium.

Title:

Determination of Trace Elements in Triglycine sulfate solutions.

Objectives:

1. To spike a triglycine sulfate (TGS) solution with 5 ppm of each of the elements Fe, Ni, Cr, Mn, Cu, Zn, Co, Pb, Cd, and Au.
2. To remove glycine from the TGS solution, followed by determining the above elements in the filtrate.
3. To recognize the elements that can be chelated by the derivative 1-naphthyl isocyanate glycine.

Introduction:

Triglycine sulfate (TGS) is an infrared detector material that has received considerable attention as a good candidate for studies of microgravity crystal growth. A space lab 3 experiment performed the growth of a TGS crystal in the fluid experiment System (FES) crystal growth cell. The extensive background on TGS consistently indicates that trace metals contaminate the saturated TGS solution regardless of initial component impurities Some of the impurities are probably the result of metals in the sulfuric acid used to synthesize the TGS crystal; others are as a result of interaction between the solution and the FES chamber itself, which is usually lined with gold foil. A vital part of the ongoing definition of crystal growth dynamics, solution quality control, and hardware optimization depends largely on the accuracy of trace metals analyses in TGS solutions.

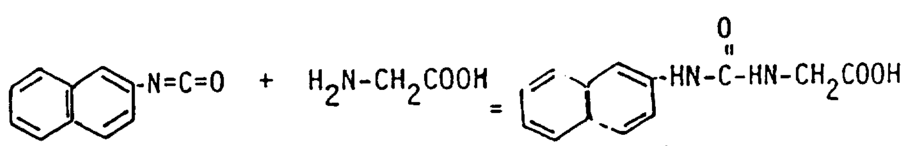
Research plan:

Two groups of metals were studied. The first group composed of iron, Fe, nickel, Ni, chromium, Cr, copper, Cu, and gold, Au. The second group composed of zinc, Zn, cobalt, Co, lead, Pb, cadmium, Cd, and gold, Au.

Five ppm of each element of each group was spiked in a 1% triglycine sulfate (TGS) solution. The mixture was put in a 100-ml polyethylene volumetric flask and filled up to the mark.

A control solution was made like before but without TGS.

The TGS in the first bottle of each group was removed by reacting[1] it with 1-naphthyl isocyanate in ether medium.



1-naphthyl isocyanate + glycine = 1-naphthyl isocyanate glycine

The derivative 1-naphthyl isocyanate glycine was removed by gravity filtration, and the filtrate was analyzed for the corresponding elements.

The elements of the control solution were also determined.

The 5100 Perkin-Elmer atomic absorption spectrophotometer, flame mode, was used for analysis purpose. The results are shown in tables 1 and 2.

Table 1: Periodical measurements of the elements in the first group in ppm unit.

Element	at once	one week	two week	three week	four week	14 weeks	24 weeks
Cu W/out TGS	5	5	5	5	5	5.0	5.1
with TGS	5	5	5	5	5	5.0	5.1
Cr w/out TGS	5	3	3	3	3	3.1	3.1
with TGS	5	3	3	3	3	3.0	3.0
Ni w/out TGS	5	5	5	5	5	5	3.1
with TGS	5	5	5	5	5	5.1	5.1
Mn w/out TGS	5	5	5	5	5	5.0	5.1
with TGS	5	5	5	5	5	5.1	5.1
Au w/out TGS	5	3	3	3	3	3.1	3.1
with TGS	5	.1	.1	.1	.1	.1	.1
Fe w/out TGS	5	5	5	5	5	5.1	5.1
with TGS	5	5	5	5	5	5.0	5.1

Table 2: Periodical measurements of the elements of the second group in ppm unit.

Element	at once	one week	two week	three week	four week	fourteen week	24 week
Co W/out TGS	5	5	5	5	5	5.1	5.2
with TGS	5	5	5	5	5	5.1	5.1
Cd w/out TGS	5	5	5	5	5	5.1	5.1
with TGS	5	5	5	5	5	5.1	5.2
Pb w/out TGS	5	5	5	5	5	5.1	5.1
with TGS	5	5	5	5	5	5.0	5.1
Zn w/out TGS	5	5	5	5	5	5.1	5.2
with TGS	5	5	5	5	5	5.1	5.1
Au w/out TGS	5	3	3	3	3	3.1	3.2
with TGS	5	0	0	0	0	0	0

Elements Behaviour in a Mixture:

Chromium atom, Cr, in the electrochemical series is considered a strong reducing agent (Table 3). Therefore, the chemical equillibrium in eq. (1) will be shifted to the right hand side:

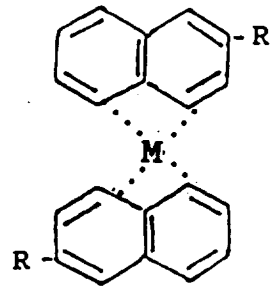
$$Cr \rightleftarrows Cr^{3+} + 3e^{-} \qquad E = -0.744 \qquad (1)$$

As a result, only 3 ppm Cr^{3+} (Table 1)of the spiked Chromium solution could be detected by the atomic absorption spectrophotometer, AAS. The rest 2 ppm were chelated by the derivative 1-naphthyl isocyanate glycine (1-NICG) (Table 1)

On the other hand gold ion Au^{3+} (Table 3) is considered a " Lewis acid", i.e. electron seeking species. It is considered a strong oxidizing agent. Therefore, the chemical equillibrium in eq.(2) is shifted to the left hand side:

$$Au \rightleftarrows Au^{3+} + 3e^{-} \qquad E = +1.498 \quad (2)$$

As a result, a small proportion, .1 ppm Au^{3+} of the spiked gold could be detected by AAS. The rest 4.9 ppm were chelated by the derivative 1-NICG (Table 1). A decrease of Au concentrtion to Zero value had been noticed after the 1st week (Table 2). The structural formula of the chelated compounds with gold or chromium can be represented by the formula indicated in figure 1. X-ray spectroscopy can be used to confirm this structural formula.



Proposed Formula for the chelated compounds

$R = NH\text{-}CO\text{-}NH\text{-}CH_2\text{-}COOH$

M = Cr, Au

(Figure 1)

Table 3

Electrochemical Series[2]

Red.	⇄	Ox.	+ ⊖	ϵ_0
Li	⇄	Li^+	+ ⊖	− 3.045[61]
K	⇄	K^+	+ ⊖	− 2.925
Ca	⇄	Ca^{2+}	+ 2 ⊖	− 2.866
Na	⇄	Na^+	+ ⊖	− 2.714
Mg	⇄	Mg^{2+}	+ 2 ⊖	− 2.363
Be	⇄	Be^{2+}	+ 2 ⊖	− 1.847
Al	⇄	Al^{3+}	+ 3 ⊖	− 1.662
Mn	⇄	Mn^{2+}	+ 2 ⊖	− 1.180
Zn (am)	⇄	Zn^{2+}	+ 2 ⊖	− 0.7627
Cr	⇄	Cr^{3+}	+ 3 ⊖	− 0.744
Ga	⇄	Ga^{3+}	+ 3 ⊖	− 0.529
Fe	⇄	Fe^{2+}	+ 2 ⊖	− 0.4002
Cd	⇄	Cd^{2+}	+ 2 ⊖	− 0.4029
Co	⇄	Co^{2+}	+ 2 ⊖	− 0.277
Ni	⇄	Ni^{2+}	+ 2 ⊖	− 0.250
Sn (weiß)	⇄	Sn^{2+}	+ 2 ⊖	− 0.136
Pb	⇄	Pb^{2+}	+ 2 ⊖	− 0.126
D_2	⇄	$2D^+$	+ 2 ⊖	− 0.0034
H_2	⇄	$2H^+$	+ 2 ⊖	∓ 0.0000
Cu	⇄	Cu^{2+}	+ 2 ⊖	+ 0.337
Ag	⇄	Ag^+	+ ⊖	+ 0.7991
Hg	⇄	Hg^{2+}	+ 2 ⊖	+ 0.854
Pd	⇄	Pd^{2+}	+ 2 ⊖	+ 0.987
Pt	⇄	Pt^{2+}	+ 2 ⊖	~+ 1.2
Au	⇄	Au^{3+}	+ 3 ⊖	+ 1.498

REDUCING AGENTS

OXIDIZING AGENTS

Extension to the Project:

Further research is needed to determine the concentration other elements in triglycine sulfate (TGS) solutions. These elements will include lithium, sodium, rubidium, magnesium, calcium, strontium, barium, aluminum, and silicon.

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References

1. Mitchell, c. A. "Commercial Organic Analysis." Vol. VIII 5th Ed. Balckiston s son and Co., Inc. Phyladelphia, 1930, p. 234

2. Holleman, A. F. and Wiberg, E. "Lerhbuch der Anorganischen Chemie." 80-90 Auflage, de Gryter: Berlin, New York, 1976, p. 202