

## A Flame Spectroscopic Study of the Stability of Dilute Solutions of Inorganic Salts

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**ABSTRACT.** Flame spectroscopy has been used to monitor net changes in concentration of dilute solution of thirty four elements at different concentrations in Pyrex, soft glass, and polyethylene containers for periods up to one year or more.

Dilute solutions are frequently unstable and may change in concentration over extended periods of time. The concentration changes are not only inconvenient because of the necessity of preparing fresh standard solutions, but are also potential serious sources of error in trace metal analysis.

Three types of processes are primarily responsible for concentration changes in dilute solutions. These are adsorption, leaching, and desorption (Minczewski 1967). Adsorption produces a loss of sample, whereas leaching of the vessel wall or desorption of a previously adsorbed sample produces an increase in concentration. These processes compete, and net change in concentration is determined by the extent to which each has occurred.

The adsorption process is the most thoroughly studied because of the ease with which it can be observed with radioactive tracers (Haissinky 1964). The extent of adsorption is dependent on the pH, the composition of the wall (soft glass, borosilicate glass, plastic, etc.), the temperature, the contact time, the solvent, and the composition of the solution (Mizuike 1965). Starik (1956) has written a comprehensive monograph on the adsorption of radioactive isotopes. The trace element composition of different containers and the leaching of trace elements in acid or basic solution have been described (Minczewski 1967, Mizuike 1965, Thiers 1957b, Eicholz *et al.* 1965, Thiers 1957a).

While adsorption losses are undoubtedly among the most important contributions to solution instability, net changes in concentration are of prime

concern to the analyst. Flame spectroscopy (emission and adsorption) offers a convenient means of monitoring net concentration changes in dilute solutions. The present study was undertaken to determine net changes in several inorganic solutions at different concentrations, in various containers, and for different periods of time up to one year or more.

### Experimental

Stock solutions of approximately 100 ppm of the elements to be studied were prepared as previously described (Christain 1968) from reagent grade chemicals. Niobium pentoxide fused with potassium carbonate and then dissolved in water. The rare earth solutions were prepared from the oxides dissolved in hydrochloric or sulfuric acid. Hydrochloric, nitric, or sulfuric acid was used to prepare other solutions as required. The stock solutions were stored in ground glass stopped Pyrex volumetric flasks. The compositions of the stock solutions are given in Table 1.

All test solutions were prepared by direct dilution of the stock solutions with water. These represented typical solutions prepared in the laboratory. Avoidance of adding more acid to the dilute solutions eliminated possible contamination from the acids. Triply distilled water was deionized with a mixed bed ion exchange column and was used for preparation of all solutions.

Solutions were analyzed by flame emission and atomic absorption spectroscopy using an Instrumentation Laboratory Model 153 Atomic Absorption Spectrophotometer. Conditions for the atomic absorption (Christian and Feldman 1970) and flame emission (Christian and Feldman 1971a, Christain and Feldman 1971b) measurements were as previously described. All test solutions were compared against dilute solutions freshly prepared from the stock solutions for instrument calibration.

### Results and Discussion

Dilute solutions of the metals were prepared by appropriate dilution of the stock solutions and were placed in new Corning Pyrex (Borosilicate glass), soft glass, and Kimble polyethylene bottles. The volume of each solution was 100 ml. The areas of the containers' surfaces exposed to the solutions were as follows; 95 cm<sup>2</sup> for the pyrex bottles, 101/cm<sup>2</sup> for the soft glass bottles and 96 cm<sup>2</sup> for the polyethylene bottles. All solutions were stored at room temperature. The results of the studies are summarized in table 2. In general, the precision of measurements was of  $\pm 5\%$  or better, and apparent changes of 5% or less are considered insignificant. Detection limits for the determinations have been

previously described (Christain 1968, Christian and Feldman 1970, Christian and Feldman 1971a). The data in the table indicate the net changes in solutions concentrations.

**Table 1.** Preparation of Stock Solutions (1000 parts per million)

Element	Source compound	Reagent added	pH of solution
Ag	AgNO <sub>3</sub>	HNO <sub>3</sub>	1.70
Al	AlCl <sub>3</sub> ·6H <sub>2</sub> O	HCl	3.39
Au	AuCl	HCl	2.50
Ba	BaCl <sub>2</sub> ·2H <sub>2</sub> O	HCl	5.95
Ca	CaCl <sub>2</sub>	HCl	2.12
Cd	CdCl <sub>2</sub> ·2½H <sub>2</sub> O	HCl	2.50
Co	CoCl <sub>2</sub> Ce(NO <sub>3</sub> ) <sub>6</sub>	HNO <sub>3</sub>	2.68
Cr	CrCl <sub>3</sub> ·6H <sub>2</sub> O	HCl	2.45
Cu	Cu(NO <sub>3</sub> ) <sub>2</sub> ·3H <sub>2</sub> O	HCl+HNO <sub>3</sub>	2.10
Dy	Dy <sub>2</sub> O <sub>3</sub>	HCl (200 ml/l)	—
Er	Er <sub>2</sub> O <sub>3</sub>	HCl (200 ml/l)	—
Fe	FeNH <sub>4</sub> (SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O	H <sub>2</sub> SO <sub>4</sub> (80 ml/l)	—
Ga	Ga metal	HCl and HNO <sub>3</sub>	0.40
Gd	Gd <sub>2</sub> O <sub>3</sub>	HCl	0.30
In	In metal	HCl	0.95
La	La <sub>2</sub> O <sub>3</sub>	HCl	1.05
Li	LiCl	HCl	5.45
Mn	MnCl <sub>2</sub>	HCl+HNO <sub>3</sub>	0.78
Mo	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>2</sub> ·4H <sub>2</sub> O		4.70
Na	NaCl		6.75
Nb	Nb <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> CO <sub>3</sub> fusion	12.50
Nd	Nd <sub>2</sub> O <sub>3</sub>	HCl (75 ml/l)	—
Ni	NiCl <sub>2</sub> ·6H <sub>2</sub> O	HCl	5.85
Pr	Pr <sub>6</sub> O <sub>11</sub>	HCl	0.10
Pt	H <sub>2</sub> PtCl <sub>6</sub> ·6H <sub>2</sub> O		2.25
Sm	Sm <sub>2</sub> O <sub>3</sub>	HCl (200 ml/l)	—
Sr	SrCl <sub>2</sub> ·6H <sub>2</sub> O	HCl	4.95
Tb	Tb <sub>4</sub> O <sub>7</sub>	HCl (250 ml/l)	—
Tl	Tl(NO <sub>3</sub> ) <sub>3</sub>		4.90
U	UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	HNO <sub>3</sub>	3.30
V	V <sub>2</sub> O <sub>5</sub>	HCl and HNO <sub>3</sub>	0.30
W	Na <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O		6.80
Y	Y <sub>2</sub> O <sub>3</sub>	HCL (30 ml/l) and H <sub>2</sub> SO <sub>4</sub> (25 ml/l)	—
Yb	Yb <sub>2</sub> O <sub>3</sub>	HCl (300 ml/l)	—

**Table 2.** Stability of Dilute Solutions

Percent element remaining after stated storage period

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
Ag	100 ppm	Pyrex					441-100
		Pyrex					441-81
	10 ppm	Soft glass	37-92	64-86	96-78	96-78	
		Polyethylene	37-95	59-92	91-87		
		Pyrex					419-6.5
		Soft glass	37-44	64-38	96-31	128-25	
1 ppm	Polyethylene	37-77	59-62	91-57			
	Pyrex					420-100	
	Soft glass	37-100				420-70	
Al	100 ppm	Pyrex					454-100
	10 ppm	Pyrex					420-100
	Soft glass	37-100					420-70
Au	100 ppm	Pyrex	37-92	95-84	91-77		441-100
		Soft glass	37-105				
	75 ppm	Soft glass	37-94	64-94			
Ba	10 ppm	Polyethylene	37-100				
		Pyrex					443-85
		Soft glass	37-96	64-100	96-97	133-104	
	5 ppm	Polyethylene	37-100	59-98	86-104		
		Soft glass	37-90	64-95	96-100	133-103	
		Polyethylene	37-100	59-105	96-114		
2 ppm	Pyrex					443-52	
	Soft glass	37-75	64-70	96-68	133-70		
	Polyethylene	37-82	59-86	96-80			

Table 2.—(Contd.)

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
Ca	1 ppm	Soft glass	37-99	64-99	96-95	135-93	
		Polyethylene	37-100	59-92	98-88		
Cd	0.1 ppm	Pyrex					419-80
		Soft glass	37-87	64-95	96-90	135-122	
	Polyethylene	37-102	59-85	98-74			
	100 ppm	Pyrex					459-87
Co	75 ppm	Soft glass	42-64	64-66	96-62	135-66	
		Polyethylene	32-100	54-99	93-100		
	50 ppm	Soft glass	42-70	64-75	96-74	135-86	
		Polyethylene	32-96	54-98	93-101		
	20 ppm	Soft glass	42-48	64-50	96-55	135-67	
		Polyethylene	32-100	54-100	93-105		
Cr	10 ppm	Soft glass	42-100	65-100	96-101	135-100	
		Polyethylene	33-100	54-102	93-100		
	5 ppm	Soft glass	42-101	65-98	96-100	135-102	
		Polyethylene	33-101	54-102	93-100		
Cu	5 ppm	Soft glass	42-94	65-95	96-95	135-102	
		Polyethylene	33-99	54-103	93-100		
Cu	10 ppm	Pyrex				445-99	
		Soft glass	40-100				
	5 ppm	Pyrex					426-176
		Polyethylene	33-101	54-100	93-93		
1 ppm	Soft glass	40-91	73-95	94-98	135-94		
	Polyethylene	33-96	54-100	93-106			
	0.5 ppm	Pyrex					426-228

Table 2.—(Contd.)

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
Dy	100 ppm	Soft glass	40-95	73-88	94-90	135-94	477-75
		Polyethylene	33-100	54-100	54-99	93-105	
		Pyrex					
		Soft glass	40-92	73-92	102-90	133-100	
Er	100 ppm	Polyethylene	33-95	54-95	93-98		477-30
		Pyrex					
		Polyethylene	33-109	54-92	43-100		
		Pyrex					
Fe	100 ppm	Soft glass	40-101				1 year - 96
		Pyrex					1 year - 100
		Pyrex					1 year - 100
		Pyrex					431-100
Ga	100 ppm	Soft glass	40-98				431-97
		Pyrex					
		Soft glass	45-100	74-99	98-100	133-100	
		Polyethylene	29-100	43-99	88-98		
Gd	100 ppm	Pyrex					431-95
		Soft glass	45-102	74-100	98-98	133-100	
		Polyethylene	29-95	53-100	88-96		
		Pyrex					
In	100 ppm	Soft glass	45-100				1 year - 100
		Pyrex					
		Soft glass	45-101				
		Pyrex					
	10 ppm	Pyrex					431-100
		Soft glass	45-101				
		Soft glass	45-101				431-106

Table 2.—(Contd.)

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
La	100 ppm	Pyrex					1 year - 90
		Soft glass	45-100	74-100	98-100	133-101	
		Polyethylene	29-100	53-98	88-103		
	10 ppm	Pyrex					1 year - 104
		Soft glass	45-102	74-95	98-100	133-100	
		Polyethylene	29-101	53-100	88-100		
Li	10 ppm	Pyrex					454-103
		Soft glass	43-97	72-101	93-100	130-100	
		Polyethylene	29-100	50-100	87-100		
	5 ppm	Soft glass	43-98	72-98	93-98	130-100	
		Polyethylene	29-100	50-102	87-100		
	0.5 ppm	Soft glass	43-102	72-103	93-98		
Polyethylene		29-100	50-95	87-101			
Mn	0.005 ppm	Pyrex					1 year - 91
	100 ppm	Pyrex					476-100
		Soft glass	43-100				
	10 ppm	Pyrex					476-103
		Soft glass	43-103				
1 ppm	Pyrex					476-96	
Mo	10 ppm	Pyrex					1 year - 133
		Soft glass	44-90	72-94	93-76	130-70	
		Polyethylene	28-99	49-107	83-100		
Na	1 ppm	Pyrex					456-132
		Soft glass	44-111	72-115	93-122	139-126	
		Polyethylene	28-98	49-100	95-88		

Table 2.—(Contd.)

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
Nb	100 ppm	Soft glass	44-100	72-100	93-98	139-100	
		Polyethylene	28-98	49-98	95-100		
Nd	100 ppm	Pyrex					1 year - 100
		Soft glass	44-100				
Ni	100 ppm	Soft glass	44-100	71-100	92-100	139-101	
		Polyethylene	28-100	49-100	96-100		
Pr	10 ppm	Soft glass	44-100	71-100	92-95	139-100	
		Polyethylene	28-99	49-94	96-97		
Pt	120 ppm	Pyrex					432-100
		Soft glass	44-100				
Sm	100 ppm	Soft glass	44-100	71-99	92-100	139-102	
		Polyethylene	44-100	49-98	96-106		
Sr	10 ppm	Pyrex					1 year - 96
		Soft glass	44-100	71-100	92-100	139-101	
Tb	10 ppm	Polyethylene	28-100	49-102	96-99		1 year - 96
		Pyrex					
Sr	10 ppm	Soft glass	44-104	71-100	92-100	139-103	
		Polyethylene	28-95	49-100	96-90		
Sr	1 ppm	Pyrex					445-104
		Soft glass	43-105	71-97	92-99	139-96	
Tb	1 ppm	Polyethylene	23-100	49-99	96-98		455-103
		Pyrex					
Tb	100 ppm	Soft glass	43-116	71-95	92-101	139-115	
		Polyethylene	28-92	49-96	96-84		
Tb	100 ppm	Pyrex					467-93
		Soft glass	43-97	71-96	92-100	139-100	



Table 2.—(Contd.)

Element	Concentration	Container	Days-%	Days-%	Days-%	Days-%	Days-%
Tl	100 ppm	Polyethylene	28-100	49-105	96-100		
		Pyrex					407-100
	10 ppm	Soft glass	43-100				
		Pyrex					407-73
U	100 ppm	Soft glass	43-90	78-100	92-102	139-100	
		Polyethylene	28-100	49-103	96-100		
	10 ppm	Pyrex					466-100
		Soft glass	43-100				
V	100 ppm	Soft glass	43-100	71-95	91-108	139-100	
		Polyethylene	28-100	49-100	96-100		
	10 ppm	Pyrex					407-92
		Soft glass	43-95	71-97	92-99	139-103	
W	100 ppm	Polyethylene	28-100	49-101	96-103		
		Soft glass	43-84	71-85	92-89		407-100
	10 ppm	Polyethylene	28-100	49-100	96-105		
		Soft glass	43-100	71-96	92-100	139-100	
Y	100 ppm	Polyethylene	28-96	49-100	96-92		
		Soft glass	43-100	71-100	92-100	139-103	
Yb	10 ppm	Polyethylene	28-100	49-100	96-100		
		Pyrex					1 year - 100
	1 ppm	Soft glass	43-100				
		Pyrex					1 year - 100
		Soft glass	43-103				

Solutions of the following elements (ppm) were found to be stable for one year or more in Pyrex containers: Cr(10), Er(1), Fe(100), Ga(10), Gd(100), In(10), La(10), Li(0.005), Nd(100), Pr(120), Sm(10), Sr(1), Tb(100), Yb(1). These were the lowest concentrations studied and may not represent the lowest stable concentrations. There appeared to have been perhaps a slight loss of Li (9%) and Tb (7%) at these concentrations. Note that even very dilute solutions of Li are stable for long periods. The following elements (ppm) were studied for shorter periods of time and their solutions were stable in soft glass containers for 135-139 days: Co(5), Nb(100), Ni(10), Pt(100), U(10), and Y(100).

No substantial changes were found for any of the above listed elements in any of the three containers for the concentrations and times studied, except for Sr where some loss appeared in polyethylene. Water hardness and extraneous ions affect Sr adsorption (Eicholz *et al.* 1965). In general, no serious changes were observed for the rare earths in any of the containers except for Dy after one year in Pyrex where losses were high; a slight loss of Tb mentioned above. Serious errors from chromium desorption are possible when a dichromate-sulfuric acid cleaning solution is used to clean the vessels (Butler and Johnston 1954). La has been reported to be absorbed from neutral solution onto plastic and to a less extent on Pyrex (Eicholz *et al.* 1965). Eighty percent of a 10 ppm Ni solution has been reported to be lost in soft glass containers in 75 days (Thiers 1957a), in contrast to the present results.

The following elements (ppm) showed no change for one year or more in Pyrex containers, although losses were observed at lower concentrations and/or with other containers: Ag(100), Al(10), Au(100), Mn(1), Sr(1) V(10). Ag at 10 ppm and Al at 1 ppm were not stable in any of the containers. The Ag adsorption losses were expected. NH<sub>3</sub> is reported to be best at stabilizing Ag solutions (Pronin *et al.* 1973); the stability in HNO<sub>3</sub> solutions depends to a large degree on the Ag(I) and HNO<sub>3</sub> concentrations. Mn and V exhibited instability in soft glass, as did Sr in soft glass and polyethylene. Thiers (1957a) reported 90% loss of Mn on soft glass in 75 days, but in the present study this concentration was stable for 43 days. A solution of 20-500 ppm reportedly exhibits good stability in 2-6 M HCl for a period of 30 days. Hydrolysis or the formation of new Au complexes are reported to have no effect in atomic absorption measurements since all Au compounds are fully dissociated in the flame (Pronin *et al.* 1973). The addition of 10% HNO<sub>3</sub> has no effect and so aqua regia can be used in Au determination.

Although Ba and Cu were unstable in Pyrex for long periods of time (> 1 yr), they were stable for shorter periods in soft glass and polyethylene: concentrations of less than 5 ppm Ba showed losses in all containers, however. W (100 ppm) was stable in soft glass for 139 days, but not in polyethylene. Cd was stable only in polyethylene.

Both Ca and Na were unstable in all the containers at low concentrations. Ca exhibited losses, except in soft glass, at greater than 90 days, while Na exhibited increases, except in polyethylene. The Na content of borosilicate glass is about 30,000 ppm (Thiers 1957a) while it is reported to be between 0.3 and 10 ppm in polyethylene (Mizuike 1965, Thiers 1957a). Cu was fairly stable in soft glass and polyethylene, although 56% of 25 ppm Cu has been reported to be adsorbed on to glass walls after 1 mo. (Gavrishin 1968). The high concentrations of Cu found for prolonged storage in Pyrex containers was surprising and it is possible contamination occurred.

Electrolytes such as NaCl and Na<sub>2</sub>SO<sub>4</sub> should reportedly be used for stabilization of Pt (IV) standard solutions (Pronin *et al.* 1973). In the present study, 100 ppm Pt as H<sub>2</sub>PtCl<sub>6</sub> dissolved in water was stable for several months in either Pyrex or soft glass containers.

Investigations have been made on the stability of solutions of several elements that were not included in this study. Valuable information can be found for the stability of solutions of As (Cheam and Agemian 1980a), Se (Cheam and Agemian 1980b, May and Kane 1984), and Ir, Os, and Ru (Gladney and Apt 1976). Numerous studies have been made on the stability of Hg solutions (Feldman 1973, Rosain and Wai 1973, Rook and Moody 1974, Moody *et al.* 1976, Newton and Ellis 1974, Avotin and Jenne 1975, Heiden and Aikens 1977, Christmann and Ingle 1976, Sanemasa *et al.* 1976, Nakayama *et al.* 1977, Ambe and Suwabe 1977, Carden 1978, Sakamoto and Kamada 1983, Zaletova 1980).

Moody and Lindstrom (1977, 1978) have investigated the selection and cleaning of plastic containers for use in trace analysis. Twelve different plastics were examined for the quantities of impurities present in the various plastics as well as the quantities of impurities leached from the plastic by said cleaning. Polyethylene and Teflon containers gave the best results in the impurity studies. The optimum cleaning procedure is leaching with 1:1 HCl-H<sub>2</sub>O for a week, rinsing with H<sub>2</sub>O, leaching with 1:1 HNO<sub>3</sub>-H<sub>2</sub>O for another week, rinsing, and filling with high-purity H<sub>2</sub>O. Loss of H<sub>2</sub>O from polypropylene and Teflon FEP containers was less than 0.1%/year. An additional moisture barrier was used with conventional polyethylene containers.

Das *et al.* (1980) have attempted to quantify the influence of wall adsorption in trace analysis, defining the retention in terms of three dimensionless parameters: surface capacity/total amount of solution, concentration of adsorbable species/total concentration, and ratio of adsorption parameters (from radiotracer measurements).

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## إستعمال أجهزة طيف الإنبعاث وطيف الإمتصاص الذري لدراسة درجة ثبات المحاليل المخففة للأملاح غير العضوية

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يتضمن هذا البحث اجراء تحليل كيميائي بواسطة طيف الإمتصاص الذري، وبواسطة طيف إنبعاث اللهب لمحاليل مختلفة التركيز، تحتوي علي مركبات كيميائية لأربع وثلاثين عنصراً، وذلك لمعرفة التغير الحاصل في تراكيزها بعد تخزينها لمدة سنة أو أكثر، في أوعية زجاج البايكس وزجاج الصوديوم (الزجاج اللين)، وأوعية البلاستيك (البوليثين).

فقد تم تحضير محاليل التخزين للعناصر التي درست بتركيز تقريبي مقداره ١٠٠ جزء من مليون وذلك بإذابة مركباتها في الماء أو الأحماض مثل حامض الهيدروكلوريك وحامض الكبريتيك وحامض النيتريك.

خزنت المحاليل في دوارق حجمية من الزجاج وأغلقت بأغطية زجاجية محكمة. تم تحضير محاليل القياس من محاليل التخزين بواسطة التخفيف المباشر بالماء. لم يضاف لها أي أحماض تجنباً لتلويثها بشوائب هذه الأحماض.

تم تحليل هذه المحاليل بواسطة أجهزة طيف الإنبعاث وطيف الإمتصاص الذري وباستعمال اللهب في الحالتين.

جميع محاليل التحليل المحضرة من محاليل التخزين قورنت نتائجها مع نتائج محاليل جديدة ومحضرة حديثاً. كذلك استعملت هذه المحاليل الجديدة لضبط الأجهزة المستعملة.

وضعت محاليل التحليل المحضرة من محاليل التخزين في قوارير من زجاج البايركس والزجاج اللين ومن البوليثلين، وخزنت في درجة حرارة الغرفة لمدد مختلفة ثم تمت عملية التحليل لمعرفة ما طرأ عليها من تغيير. وقد وُضحت نتائج التحليل في الجدول رقم (٢) من الدراسة. جميع التغيرات في التركيز والتي تقل عن ٥٪ أهملت، واعتبرت فقط التغيرات التي تزيد على هذه القيمة. كانت درجة الضبط في القياسات أقل من  $\pm ٥\%$ .

وجد أن محاليل العناصر التالية المحفوظة في أوعية من زجاج البايركس تتمتع بثبات لمدة تزيد على السنة، وهي: الكروم والحديد والجاليوم والجادلينيوم واليوربيوم والأنديوم واللانثانيوم والليثيوم والنيوديميوم والبرازيوديميوم والسماريوم والسترانشيوم والتيريبيوم واليتربيوم، وذلك على مدى من التركيز يختلف من عنصر إلى آخر، وكذلك كانت النتيجة في بقية أنواع الأوعية المستعملة وللمدة نفسها ما عدا السترنشيوم الذي عانى بعض النقص عندما خزن في أوعية البوليثلين.

وقد تم في هذه الدراسة إثبات درجة ثبات بقية العناصر في أوعية التخزين المختلفة ومدد مختلفة حددت جميعها في متن الدراسة.