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A COMPARISON OF VARIOUS METHODS

OF ASSAYING CYANIDE SOLUTIONS FOR GOLD $\mathcal{T22/}_{BY}$

THEODORE SAUNDERS DUNN

A THESIS FOR THE DEGREE

 $\mathbf{0}\mathbf{F}$

BACHELOR OF SCIENCE IN GENERAL SCIENCE

MISSOURI SCHOOL OF MINES AND METALLURGY

ROLLA, MISSOURI

MAY, 1910

O.K. H. Manno

10924

A COMPARISON OF VARIOUS METHODS OF ASSAYING CYATIDE SOLUTIONS FOR GOLD.

Although there has been a great deal written on the different methods of assaying cyanide solutions and new methods are being devised constantly there has been very little said as to the relative merits of the methods in use. An attempt has therefore been made in this paper to compare several of the methods now used.

The method of procedure was as follows:-Four cyanide solutions were prepared of the following strengths and richness:

Solution #I	0.5 % KCN.	I.oz. Au. per ton.
Solution #2	0.5 % KCN.	.05 oz. Au. per ton.
Solution #3	0.05 % KCN.	Loz. Au. per ton.
Solution #4	0.05 % KCN.	.05 oz. Au. per ton.

The gold was weighted out and then put into the form of gold chloride and then into the cyanide solutions. The solutions were assayed by taking five samples of each solution and then carrying the twenty assays thus taken through all the processes, comparing each method as to accuracy, speed and simpleness. Each process was run through several times to gain familiarity and speed before the time was taken.

The following methods were used in the work:-

Method I - Evaporating in a lead dish.

Method 2 - Chiddey's Method.

Method 3 - Evaporating to small bulk in an evaporating dish and absorbing the remainder with lithagge.

Method 4 - Evaporating to small bulk in an evaporating dish and absorbing the remainder with litharge and silica.

Method 5 - Miller's method of precipitating with powdered copper sulphate.

Method 6 - Lindeman's Method of precipitating with ammoniacal copper nitrate.

Method 7 - Arent's method of precipitating with cement copper.

Method 8 - Del Mar's method of precipitating with aluminuím sulphide.

Method 9 - Precipitation with silver nitrate. Method IO - Mohr's Colonimetric Method. Method II - Seamon's method of precipitating with aluminum foil.

Of the rich solutions one assay ton was taken and of the poor solutions ten assay tons.

The results obtained were as follows:-

Method I-

Evaporation to dryness in lead dish. The dish was was folded up and cupelled.

Solution I.

0.5 % KCN. I oz. Au. per ton.

Sample-Assay oz per ton-Actual loss in oz per ton-% loss

Ι	•99	.01	I%
2	•99	.01	I%
3	. 995	.005	.5%
4	• 995	.005	. 5 %
5	.995	.005	.5%
Average	.993	.007	.7%
m :	Dift		

Time - Fifty minutes.

	Scluti	on 2.	
	.5 % KCN.	.05 oz. Au. per	• ton
Sample-Ass	ay oz per ton-Act	ual loss oz per	ton-% loss.
I	.04 8	.002	4 %
2	.04 85	.0015	3 %
3	.049	.001	2 %
4	.0 49	.001	2 %
5	.049	.00I	2 %
Average	.0487	.0013	2.6 %
Time	- One hour, thirt	y minutes.	
	Solution	<i>х</i>	
		I oz. Au. per t	(A)
Comple-Auc	ay oz per ton-Act		
I I	.99	.0I	I %
2	.995	,00 5	5%
2 3	.99	.01	I %
4	.99	.01	I %
5	.99	.01	I %
Average	•99 I	.009	.9 %
m)			

Time - Fifty minutes.

	Solution	4.	
	.05 % KCN.	.05 oz. Au. per	ton
Sample-	Assay oz per ton-	Actual loss oz per	ton-%loss
I	.049	.001	2 %
2	. 049	•00I	2 7
3	,0485	.0015	3 %
4	₽04 85	.0015	3 %
4	.049	.001	2 %
Average	.04 88	.0012	2.4 %

Time - One hour, thirty minutes.

Buttons all cupelled well and rapidly but in the case of a poor solution the time of evaporation was greatly lengthened and so caused the time of operation to become somewhat long.

Method 2.

Chiddey's Method.

Solution I.

.5 % KCN. I oz. Au. per ton

Sample-	Assay oz per ton-	Actual loss oz per	ton-%loss
I	.985	.015	I.5 %
2	•99	.01	I %
3	. 985	.015	I.5 %
4	•99	.01	I %
5	.985	.015	I.5 %
Average	.987	.013	I.3 %

Time - Fifty-five minutes.

	-	0-	
	S	olution 2.	
	.5 % KCN.	.05 oz. Au. per ton	
Sample-	Assay oz per ton	- Actual loss-in oz per	ton- $\%$ loss
I	.049	.001	2 %
2	.047	•003	6 %
3	.04 8	.002	4 %
4	.04 8	.002	4 %
5	.048	.002	4%
Av er a ge	.0 48	.002	4 %
Tir	ne - One hour, ter	n minutes.	
	Solutio	on 3.	
	.05 % KCN.	I oz. Au. per ton.	
Sample-	Assay oz per ton-	- Actual loss in oz per	ton= % loss
I	.86	.14	$\mathtt{I4}^{s}$
2	.87	.13	13%
3	•88	.12	12%
4	. 85	.15	I5 %
5	.86	.14	I 4%
Average	.864	I3. 6	I3.6 %
m:	- Difty-fine wire	at com	

Time Fifty-five minutes.

These results were obtained with a weak solution which was not brought up to strength as recommended. When the solution was brought up to about .5 % KCN. by adding some fresh KCh. the results obtained were much better and were as follows: >

Sample- Assay oz per ton- Actual loss in oz per ton- % loss

I	•98	.02	2 %
2	•99	.01	I %
3	•99	•0I	I %
4	.9 8	.02	2 %
5	.99	10.	I %
Average	98.6	$I_{\bullet}4$	I. 4 %

Time - Fifty-five minutes.

Thus showing that it is necessary in using this method to bring up the strength of a weak solution.

Solution 4.

.05 % KCN. .05 oz. Au. per ton

Sample-	Assay oz per ton-	- Actual loss in or per	r ton-%loss
I	.0 48	.002	4 %
2	.0 49	•00I	2 %
3	.04 8	.002	4~%
4	.04 8	.002	4 %
5	.049	.001	2 %
Average	.0484	.0016	3.2%

Average

•00

Time - One hour, ten minutes.

The results were as a whole good but some showed zinc when being gupelled. This was undoubtedly due to too much haste in removing the samples from the hot plate. Method 3.

Evaporate to small bulk, absorb with litharge, fuse and cupel.

Solution I.			
	.5 % KCI	N. I.oz. Au. per to	n
Sample-	Assay oz per ton-	Actual loss oz perton	- % loss
İ	.995	. 005	• 5%
2	•995	.00 5	•5 %
3	•995	.00 5	• 5%
4	•995	.005	•5%
5	. 99	.01	• 1 %
Average	.99 4	.00 6	•6 %

Time-Two hours.

Solution 2.

.5 % KCN. .05 oz. Au. per ton. Sample-Assay oz per ton-Actual loss in oz per ton-% loss 2% .00I Ι .049 2 % 2 .049 .00I 2% .049 .00I 3 4% .002 **.0**48 4 2 % .00I 5 ,049 2.4 % ,0488 .0012 Avorage

Time - Two hours, thirty minutes.

Solution 3.

.05 % KCN. I oz. Au. per ton

Sample- Assay oz per ton- Actual loss in oz per ton- % loss

Average	, 991	.09	.9 %
5	.995	.005	.5 %
4	•99	.01	I %
3	.99	•0I	I %
2	.99	•0I	I %
I	.99	.01	I %

Time - Two hours.

Solution 4.

.05 % KCN. .05 oz. Au. per ton.

Sample- Assay oz, per ton- Actual loss in oz per ton- % loss

Ι	.0 49	.001	2 %
2	.04 8	.002	4 烯
3	.04 8	.002	4 %
4	•048	.002	4 %
5	.048	.002	4 %
Average	.0 482	.0018	3.6 %

Time - Two hours, thirty minutes.

The results obtained by this method were excellent but the method is very long. The time necessary to mix charges and to fuse causing a big increase in the time necessary. Method 4.

Evaporate to small bulk, absorb with litharge and silica, fuse and cupel.

Solution I.

.5 % KCN. I oz. Au. per ton.

Sample - Assay oz per ton - Actual loss in oz per ton - % loss

I	.99	.01	I %
2	.9 95	.0 05	.5 %
3	.995	. 005	•5 %
4	.99	.01	I %
5	.99	10.	I %.
Average	992	• 0 08	•8 %

Time - Two hours.

Solution 2.

.5 % KCN. .05 oz. Au. per ton. Sample - Assay oz per ton - Actual loss in oz per ton - % loss 4% .002 Ι .048 4% .002 .048 2 2 % .00I .049 3 2 % .00I .049 4 4% .002 .048 5 3.2 % .0484 .0016 Average Time - Two hours, thirty minutes.

	-11-		
	Solution	3.	
	.05 % KCN. I oz.	. Au. per tom.	
Samp]	e- Assay oz. per ton-Actu	al loss in oz per t	ton- %loss.
I	. 995	.005	.5 %
2	. 995	• 00 •	.5 %
3	•9 9	.01	I %
4	•99	.01	I %
5	.99	.01	I
Avera	_ഋ ം 992	.008	.8%
	Time; Two hours.		,
	Solution 4.		
	.05 % KCN05	oz. Au. per ton.	
Sampl	e- Assay oz per ton- Actu	al loss in oz per t	on- %1058
I	.0 48	.002	4 %
2	.04 8	.002	4 %
3	.04 8	.002	4 %
4	.04 8	.002	4 %
5	.049	.001	2 %
Avera	ge .04 82	.00I8	3.6 %
Time - Two hours, thirty minutes.			

No difference could be noted between this method and Method 3. The results were equally as good and are open to the same objection - too long to run.

Met	hod 5 - Mille	er's Method.	
		Solution I.	
	0.5 % KCN.	I oz.Au. per ton.	
Sample-A	ssay oz.per t	ton-Actual loss in oz. per to	on-%loss.
I	.992	.008	.8 %
2	.992	•008	.8 %
3	.99	10.	I %
4	.992	.008	.8 %
5	.992	.008	.8 %
Average	.9912	.0084	.84 %
Time - Two hours, thirty minutes.			
	Solu	ation 2.	
0.	5 % KCN.	.05 Mz. Au. per ton.	
Sample-A	ssay oz. per	ton-Actual loss in oz.per to	on-% loss.
I	.049	.001	2 %
2	.049	.001	2 %
3	.04 8	.002	4%
4	.04 8	.002	4 %
5 _	.048	.002	4%
Average Tim	.0484 e - Two hours	.0016 s, fifty minutes.	3.2

Time - Two hours, fifty minutes.

Solution 3.	So	lu	ti	on	3.
-------------	----	----	----	----	----

.05 % KCN. I oz. Au. per ton Sample-Assay oz.per ton-Actual lossin oz.per ton- % loss. Ι .99 Ι% .0I .99 .0I I % 2 .992 3 .8 % .008 .99 4 .0I Ι% .992 .8 % 5 .008 .9908 Average .0092 .92 %

Time - Two hours, thirty minutes.

Solution 4.

0.05 % KCN. 0.05 oz. Au. per ton.

I .048 .002	ton- $\%$ loss.
	4 %
2 .049 .00I	2 %
3.048.002	4 9 ⁷
4 .048 .002	4 %
5 .048 .002	4 %
Average .0482 .0018	3.6 %

Time - Two hours, fifty minutes,

The results obtained from this method were fairly accurate but the .05 % KCN. solution had to be brought

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up to at least .025 % KCN. before the lusox would precipitate the values from it. Also the time for assaying was quite long.

Method 6 - Lindeman's Method.

Solution I.

0.5 % KCN. I oz. Au. per ton.

Sample-Assay oz.per ton-Actual loss in oz.per ton- % loss.

5	.982	, 93 8	I.8 %
4	•984	.016	I.6%
3	.982	.018	I .8 %
2	.982	.018	I.8%
I	.98	.02	2 /

Time - Two hours, fifty-five minutes.

Solution 2.

0.5 % KCN. 0.05 oz. Au. per ton. Sample-Assay oz.per ton-Actual loss in oz.per ton-% loss 4% .002 Ι .048 6 % .003 .047 2 6 % .047 **.0**03 3 6 % .003 .047 4 6 % **.0**03 .047 5 5.6 % .0472 .0028 Average Time - Three hours, fifteen minutes.

	Solution	. 7	
	Solutior 0.05 % KCN.	-	on.
Sample-	Assay oz.per ton-Ac		
I	. 98	.02	2 %
2	.9 8	.02	2 %
3	.97 8	.022	2.2%
4	. 978	.22	2.2%
5	.9 8	.02	2 %
Average	.97 92	.02 08	2.08 %
Tir	ne - Two hours, fif	ty-five minutes.	
	Solution	4.	
	0.05 % KCN.	0.05 oz. Au. per	ton.
Sample-A	Assay oz.per ton-Ac	tual loss in oz per	ton- 🖇 loss
I	.044	. 00 6	I 2 %
2	.045	.005	I0 %
3	.04 6	.004	8 %
4	.047	.003	6 %
5	.046	.004	8 %
Average	.04 56	.0044	8.8%

Time - Three hours, fifteen minutes.

The attrage results from this method were lower than the average from other methods and the time necessary was very long. The buttons all cupelled well and there was no trouble from copper but for some reason all results obtained were low.

Method 7 - Arent's Method.

Solution I.

	0.5 % KCN.	I oz. Au. per ton	l.
Sample-A	ssay oz.per ton	Actual loss in oz. per ton	-% 1088.
I	. 984	.016	I.6 %
2	. 984	.016	I.6 %
3	.9 86	.014	I.4%
4	.982	.018	I.8%
5	.984	.016	I.6%
Average	.9 84	.016	I.6%
Time - Two hours, fifty minutes.			
	Solut	ion 2.	
	0.5 $\%$ KCN.	0.05 oz. Au. per ton.	
Sample-A	ssay oz per tor	Actual loss in oz.per ton-	% loss.
I	.048	002	4 %
2	. 049	•001	2 %
3	.048	.002	4 %
4	.048	.002	4 🥍
5	.048	.002	4 %
Average	.0482	•0018	3.6 %
Tim	e - Three hours	s, ten minutes.	

	Sol	lution 3.	
0	.05 % KCN.	I oz. Au. per ton.	
Sample-	Assay oz.per	ton-Actual loss in oz.per	ton-% loss
I	. 984	.016	I.6 %
2	•98	.02	2 %
3	. 982	.018	I.8 %
4	.982	.018	I.8 %
5	.982	.018	I.8 %
Average	.982	.018	I.8 %
Tin	ne - Two hour	s, fifty minutes.	
	Solut	ion 4.	
C	0.05 % KCN.	.05 oz. Au. per ton.	•
Sample-A	Assay oz.per	ton-Actual loss in oz.per	ton- $\%$ loss.
I	. 48	.002	4 %
2	. 48	.002	4 %
3	.4 8	.002	4 %
4	. 48	•002	4 %
5	. 49	.001	2 %
Average	. 482	.0018	3.8 %

Time - Three hours, ten minutes.

The results obtained by this method were low and the methods were very long. The cupellation was good and no trouble of any kind experienced.

Method 8 - Del Mar's Method. Solution I. 0.5 % KCN. I oz. Au. per ton. Sample-Assay oz.per ton-Actual loss in oz.per ton-% loss 0.4% **.99**6 .004 Ι 0.2% .998 .002 2 0.4% .004 3 .996 0.4% .004 .996 4 0.4 % .996 .004 5 0.36 % .0036 Average .9964 Time - Two hours, ten minutes. Solution 2. 0.05 oz. Au. per ton. 0.5 % KCN. Sample-Assay oz.per ton-Actual loss in oz.per ton-% ton. 4% .002 .048 I 4% .002 2 .048 4 % .048 .002 3 2% .00I .049 4 4% .048 .002 5 3.6 % .0018 Average .0482 Time - Two hours, ten minutes.

	Soluti	ion 3.	
	0.05 % KCN.	I oz. Au. per to	on.
Sample-A	ssay oz. per t	on-Actual loss in oz.per	ton- 🖉 loss
Ι	.996	.004	0.4 %
2	.9 96	.004	0.4 %
3	.99 6	•00 4	0.4 %
4	.9 96	. 004	0.4 %
5	.998	.002	0.2 %
Average	.99 64	. 99 ³⁶	0.36%
Tim	e - Two hours,	ten minutes.	
	S ol utio	n 4.	
	0.05 % KCM.	0.05 oz. Au. per	ton.
A-elqma?	ssay oz.per to	n-Actual loss in oz.per	ton-% loss
I	.04 8	.002	456
2	.0 48	.002	4 %
3	.04 8	. 002	4 %
4	.049	•001	2 %
5	.049	100	2 %
Average	.0 484	.0 016	3.2%

Time - Two hours, ten minutes.

The results of this method were accurate and no trouble was experienced during the manipulation. The time necessary to filter and fuse added materially to the length of the assay.

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The aluminum sulphide was prepared by fusing P5S with aluminum foil in the muffle of the assay furnace and took but a short time.

Method 9 - Precipitation with silver nitrate.

Solution I.

0.5 % KCN. I oz. Au. per ton.

Sample-Assay oz.per ton-Actual loss in oz.per ton- % loss

3 .994 .006	0.56 %
3 .994 .006	0.4%
	0.4%
2 .994 .006	0.4 %
	0.4 %
I •996 •004	0.6%

Time - Two hrs, fifty minutes.

Solution 2.

0.5% KCN. 0.05 oz. Au. per ton.

Sample- Assay oz.per ton-Actual loss in oz.per ton- % loss.

Time	- Two hours,	thirty minutes.	
Average	.0482	.0018	3.6%
5	.049	.001	2 %
4	.048	.002	4 %
3	. 048	.002	4 %
2	.048	.002	4 %
I	.04 8	.002	4 %

		olution 3.	
	0.05 %	- KCN. I oz. Au. pe	r ton.
Sample-As	say oz. per	ton-Actual loss in oz.pe	r ton-% loss
I	.99	.01	I
2	.992	.008	.8 %
3	.992	. 008	.8 %
4	99	.01	I %
5	.9 9	10.	I %
	0000	H P + P + N N	60 60 6 6 6 6 6
Average		.0092	0.92 %
Time	- Two hours	s, fifty minutes.	
	Solu	ation 4.	
0.0	5% KCN.	0.05 oz. Au. per ton.	
		con-Actual loss in oz.per	ton- % loss.
I	.048	.002	4 %
2	.048	.0 02	4 %
3	.04 8	.002	4 %
4	.047	.003	6 %
5	.0 48	.002	4 %
Average	.04 78	.0022	4.4%
Average	.0478	.0022	4.4 %

Time - Two hours, thirty minutes.

The results from this method were good but the method is entirely too long. The time necessary to filter being exceptionly long, especially in the case of rich solutions. Method IO - Mohr's Colorometric Method. The results obtained from this method were so unreliable that after repeated trials it was evident that in the hands of an unexperienced operator the method was useless.

Method II - Seamon's Method. With Seamon's method no satisfactory results could be obtained. The precipitate formed rapidly but could not be washed from the aluminum foil and after repeated attempts with no different results the method was abandoned.

Conclusions:-

In choosing a method of assaying two vital qualities must necessarily be taken into consideration, those of accuracy and speed. These two qualities would of course be affected by the person using the method, though if correctly performed the accuracy of the assay would be less affected than the speed, which would vary according to the person making the assay and the conveniences for rapid work he had at his deposal. But in all assays accuracy is the important thing, so the results of my work will first be compared as to accuracy and then as to speed of performance.

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	The ave	rage res	ults o	f the a	BBAYB	run by	ach me	th od					
are:-													
Method- Sol.I-% loss-Sol.2-% loss-Sol.3-% loss-Sol.4-% loss.													
I	.993	0.7 %	.0487	2.6 %	.9 91	0.9 %	.048 8	2.4 %					
2	. 987	I.3 %	.048	4%	.9 86	I .4 %	.0484	3.2 %					
3	.994	0.6%	.0488	2.4 %	.99I	0.9 %	.0482	3.6%					
4	.992	0.8%	.04 84	3.2 %	.992	0.8 %	.0482	3. 6 %					
5	.9912	0.84 %	.0484	3.2 %	.9908	0.92 %	.0482	3.6 %					
6	.982	I.8 %	.0472	5.6%	.9792	2. 08 9	7. .045 6	8.8 %					
7	.984	I.6 %	.0482	3. 6 %	.982	I.8 %	.0482	3. 6 %					
8	.9964	0.36 %	.0482	3.6 %	,99 6 8	0.36 9	6 .0484	3.2 %					
9	,99 44	0.56 %	.0482	3.6%	.99 08	0.92 9	6 .0478	4.4 %					
10	-	-	-	-	-	-	-	-					
TT	-	-	-	-	-	-	-	-					

II m With the exception of one or two these results check fairly closely and the variation is probably due to the manipulation by the assayer and those giving the higher percentage of loss would probably give better results in the hands of a more experienced person.

In the matter of time necessary for the pperation the methods varied widely, some of them taking so long as to be impracticable when many assays are to be made or quick results are required, as will be seen by the following table:-

	SolutionI - Solution 2 - Solution 3 - Solution								
Method.	Hrs.	Min.	Hrs.	Min.	Hrs.	Min.	Hrs.	Min.	
I	0	50	I	30	0	50	I	3 0	
2	0	55	Ι	10	0	55	I	10	
3	2	0 0	2	3 0	2	00	2	3 0	
4	2	00	2	3 0	2	00	2	30	
5	2	3 0	2	5 0	2	30	2	50	
6	2	55	3	I5	2	5 5	3	I 5	
7	2	5 0	3	10	2	50	3	10	
8	2	10	2	10	2	10	2	10	
9	2	50	2	30	2	50	2	30	
10	-		-		•		-	••	
II	-		-		-	-	-		

Dy this table it will be seen that in a case where the time factor must always be taken into consideration only two methods give the requisite speed, Chiddey's and that of evaporating in a lead dish, and in the case of a poor solution Chiddey's is the more rapid, although it did not give quite so good results for me as did the evaporation in the lead dish.

When taking into account both speed and accuraty these two methods seem far ahead of the others, but it is altogether likely that in the case of making a number of assays each day a person using one of the others methods would as he became more and more proficient in the method cut down the time required to quite an extent.

So, judging from the results obtained during the work I would say that it is largely a matter of individual taste and the amount of time available as to which method would be used.

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