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THE QUANTITATIVE DETERMINATION OF GOLD, ESPECIALLY IN ANIMAL TISSUE.[1]

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In connection with the experimental work carried on by Dr. Lydia M. DeWitt on the treatment of tuberculosis with various salts of gold, it was desired to determine the distribution of the gold in the organs of the guinea pigs used in the experiments. Since the dosage of gold was small, it was necessary to find a method which would determine accurately small fractions of a milligram of gold. Heubner[2] used a method for the determination of gold in animal tissue, but the amounts were always larger than those used in our experiments and only one test experiment is recorded.

The well-known assay method has been used for determining small amounts of gold, but was found unsatisfactory in our work because of the different class of impurities in our materials. Most of the published

[1] Part of the material in this paper has been embodied in a thesis presented to the Faculty of the University of Chicago by Sidney M. Cadwell in partial fulfillment of the requirements for the degree of Doctor of Philosophy, and part of the material will be used in a similar thesis by Gladys Leavell.

[2] *Arch. exp. Path. Pharm.*, **56**, 370.

methods for the quantitative separation and determination of gold depend on the great ease with which gold salts are reduced. In some cases the amount of reagent required to reduce the gold can be accurately determined. Utilizing this principle, Gooch and Morley have developed a method which is accurate to 0.005 mg. C. H. Christmann tried to apply this method in our problem but found that the ferric iron, as well as gold, oxidized the reagent. The possibility of the presence of other and varied oxidizing or reducing substances in the solutions form organic materials made it seem wise to choose a method which did not depend on oxidation by gold. The "Purple of Cassius" method, by which gold may be estimated in dilutions as great as one part to four million, was also found unsuitable for our purpose, since, unless the gold is first purified, the colors are not stable enough for comparison. Sarah Miller[1] has developed an electrolytic method for the separation of gold from ferric iron in phosphoric acid solution, but she was unable to deposit the last few milligrams of gold in the presence of iron until the voltage was raised to 2.7, at which point some copper is also deposited, if present. She also reports some difficulty with non-adherent deposits. Gold[2] has been electrolyzed from solutions containing it in the form of a complex cyanide, thioaurate, chloride and thiocyanide and also in the presence of free phosphoric acid. Neither potassium cyanide nor sodium sulfide solutions have been used for the electrolytic separation of gold from ferric iron because they precipitate the iron.

Of all the methods published the electrolytic method seemed most suitable for our work, but it was necessary to modify Miller's procedure, since we were working with much smaller amounts which would not be completely deposited by her method. Our gold, instead of being in the form of a chloride, was fulminating gold, formed in the course of dissolving the metal after the destruction of the organic matter. In our solution, also, some copper and iron were frequently, if not always, present. The conditions of the experiment require that the solution cannot be much more concentrated than one part of gold to 20,000 parts of water. Allamand states that the excellence of an electrolytic deposit is directly proportional to the concentration of the metal ions and inversely proportional to the current density within certain limits. To offset this large dilution, therefore, the rotating anode was used, thus maintaining the concentration immediately around the cathode; we thus not only improved the deposit, but also decreased the time required for deposition. The optimum temperature of about 60° and a current density of $N.D._{100}$ = 0.07 were used. Our standard gold solution was prepared by dissolving

[1] THIS JOURNAL, **26**, 1268 (1904).

[2] Edgar F. Smith, "Electro Analysis," 5th ed., 1912; Allamand, "Electro Chemistry."

a known amount of pure gold in aqua regia. The excess of free chlorine was always removed by treatment with ammonia, thus forming fulminating gold before electrolysis.

In Table I, which summarizes the results of all the experiments made to test the method, the time is expressed in minutes and the weights in milligrams. The volume of the electrolyte was about 40 cc. and phosphoric acid and sodium phosphate were always added in the proportions prescribed by Miller. The temperature was 50 to 70° except in Expt. 1, when it was 34°. (See detailed description of method.)

TABLE I.

Summary of Test Experiments.

No.	Material analyzed.	Volts.	M. amp.	Time.	Au taken. Mg.	Au found. Mg.	Error. Mg.
1	Water	2.6	20	60	4.58	4.60	0.02
2	3 g. heart	1–1.5	2–8	105	2.28	2.26	0.02
3	5 g. kidney	1–1.5	2–10	105	2.28	2.45	0.17
4	10 g. liver	1–1.5	1–4	105	2.28	2.24	0.04
5	10 g. liver	1–1.5	2–7	105	2.28	2.29	0.01
6	$(NH_4)_2SO_4$	1.2	...	15	2.69	2.35	0.34
7	$(NH_4)_2SO_4$	1.2	...	30	2.69	2.72	0.03
8	$(NH_4)_2SO_4$	1.2	...	45	2.69	2.70	0.01
9	$(NH_4)_2SO_4$	1.2	...	60	2.69	2.79	0.10
10	$(NH_4)_2SO_4$	1.2	...	75	2.69	2.72	0.03
11	$(NH_4)_2SO_4$	1.2	...	90	2.69	2.74	0.05
12	8 g. spleen	1.2	...	10	2.69	1.89	0.80
13	4.5 g. kidney	1.2	...	20	2.69	2.43	0.26
14	7.31 g. lungs	1.2	...	30	2.69	2.68	0.01
15	19 g. liver	1.2	...	33	2.69	2.75	0.06
16	Feces	1.2	...	40	2.69	2.65	0.04
17	Urine	1.2	...	50	2.69	2.65	0.04
18	$(NH_4)_2SO_4$ 1.5 g. $(NH_4)Cl$ 1 g.	1.2	...	35	2.69	2.70	0.01
19	$(NH_4)_2SO_4$ 1.5 g. $(NH_4)Cl$ 1 g.	1.2	...	35	2.69	2.73	0.04
20	$(NH_4)_2SO_4$ 3 g. $(NH_4)Cl$ 2 g.	1.2	...	35	2.69	2.73	0.04
21	$(NH_4)_2SO_4$ 3 g. $(NH_4)Cl$ 2 g.	1.2	...	35	2.69	2.67	0.02
22	$(NH_4)_2SO_4$ 1.5 g. $(NH_4)Cl$ 1 g.	1.2	...	35	2.69	2.75	0.06
23	Copper 5 mg. Iron 5 mg.	1.2	...	35	2.69	2.72	0.03
24	$(NH_4)_2SO_4$ 1 g.[1]	1.1	...	40	2.69	2.73	0.04
25	$(NH_4)_2SO_4$ 1 g.[1] $(NH_4)Cl$	1.1	...	40	2.69	2.65	0.04
26	$(NH_4)_2SO_4$ 1 g.[2]	1.1	...	40	2.69	2.60	0.09
27	$(NH_4)_2SO_4$ 1 g.[2]	1.1	...	40	2.69	2.77	0.08
28	$(NH_4)_2SO_4$ 1 g.[3]	1.1	...	40	2.69	2.68	0.01
29	$(NH_4)_2SO_4$ 1 g.[3]	1.1	...	40	2.69	2.62	0.07

[1] Basic. [2] Neutral. [3] Acid.

TABLE I (*continued*).

No.	Material analyzed.	Volts.	M. amp.	Time.	Au. taken. Mg.	Au. found. Mg.	Error. Mg.
30	5 g. liver	1–1.5	2–11	90	5.39	5.37	0.02
31	5 g. liver	1–1.5	2–14	90	5.39	5.43	0.04
32	4 g. liver	1–1.3	0.8–5	90	2.69	2.70	0.01 0.37%
33	10 g. liver	1–1.5	1.5–6	90	2.69	2.73	0.04 1.48%
34	10 g. liver	1–1.3	0.2–0.7	90	2.69	2.59	0.10 3.7%
35	10 g. liver	1–1.3	0.7–0.8	90	2.69	2.58	0.11 4.07%
36	10 g. liver	1–1.3	0.7–2	90	0.23	0.21	0.02 8.7%
37	10 g. liver	1–1.3	0.5–2	90	0.23	0.23	0.0 0.0%
38	10 g. liver	1–1.5	0.3–2	90	0.23	0.21	0.02 8.7%
39	10 g. liver	1–1.5	1.8–2	90	0.23	0.21	0.02 8.7%
40	15 g. liver	1–1.2	1.5–2	100	0.23	0.19	0.04 17.4%
41	15 g. liver	1–1.2	4–11	100	2.69	2.70	0.01 0.37%
42	10 g. liver	1–1.2	1.5–5	100	2.69	4.60	1.91 71.0%
43	$(NH_4)Cl$ $(NH_4)_2SO_4$	1–1.5	0.7–9	90	5.39	5.37	0.02 0.37%
44	$(NH_4)Cl$ $(NH_4)_2SO_4$	1–1.5	0.7–7	90	5.39	5.48	0.09 1.7%
45	10 g. liver	1.2	...	40	0.027	0.02	0.007
46	10 g. liver	1.2	...	40	0.054	0.06	0.006
47	10 g. liver	1.2	...	40	0.08	0.09	0.01
48	10 g. liver	1.2	...	40	0.11	0.12	0.01
49	10 g. liver	1.2	...	40	0.13	0.08	0.05
50	10 g. liver	1.2	...	40	0.00	0.01	0.01
51	Au(CN)	1.2	...	40	1.97	1.94	0.03
52	Au(CN)	1.2	...	40	1.97	1.95	0.02
53	$KAu(CN)_2$	1.2	...	40	2.61	2.60	0.01
54	$KAu(CN)_2$	1.2	...	40	2.61	2.66	0.05

Expts. 1 to 5 demonstrate that the method is applicable to our conditions. In Expts. 6 to 11, inclusive, the time of electrolysis was varied for a solution of gold chloride, ammonium chloride, ammonium sulfate and the usual phosphoric acid and sodium phosphate. In Expts. 12 to 17, inclusive, the gold chloride was added to a tissue solution and the time varied. Since fulminating gold was used in our actual experiments, we insured its formation in Expts. 18 to 23 by using the same

operations as in our regular analyses. Equivalent amounts of copper and iron were added in Expts. 22 and 23. In every case, as can be seen from the table, deposition was complete in 35 minutes.

In most of the experiments after deposition was complete the current was stopped and the electrodes were removed from the solution without siphoning off the electrolyte as is customary. To prove that this caused no appreciable error, two gold-plated electrodes were left in two phosphate solutions for two minutes, with losses of only 0.01 and 0.00 mg. In a similar experiment, 0.1 mg. was lost in a half-hour and only 0.25 mg. in 4.5 hours.

Since the solutions from organic materials may contain varying amounts of iron and copper, an experiment was run testing the deposition of iron from a tissue solution containing 0.01 g. pure ferric chloride. The method of analysis was the same as used with the gold and the gain in weight of the electrode was only 0.04 mg., an error easily accounted for by incomplete washing. In two experiments 0.05 g. copper sulfate was added to a tissue solution and an ammonium salt solution, respectively. No deposition occurred at 1.2 volts, but if the voltage was a few tenths higher the deposition was rapid; this copper deposit dissolved quickly, however, when the current was stopped.

To determine whether variations in acidity of the electrolyte caused variations in the result of the analyses, the solution in Expts. 24 and 25 had a strong odor of ammonia and was electrolyzed after the addition of one and one-half times the usual amount of sodium phosphate to make up for the absence of phosphoric acid. In Expts. 26 and 27, the solution was as nearly neutral as possible, no phosphric acid having been added. The solution in Expts. 28 and 29 was unusually acid, as 2 cc. of conc. hydrochloric acid was added after the usual preparatory steps were complete. As will be seen from the table, this varying basicity or acidity had very little effect on the deposition of the gold.

Having proved that gold can be determined quantitatively in solutions derived from the decomposition of tissues and that varying amounts of iron and copper do not interfere, 10 g. of guinea-pig liver containing gold was decomposed with sulfuric and nitric acids. As neutralization of the sulfuric acid loaded our solution unduly with salts, the sulfuric acid was evaporated from the Kjeldahl flask with the aid of a gentle current of air. Part of the gold was then in metallic form and was dissolved by the addition of aqua regia, the excess of free chlorine thus formed being removed by the addition of ammonium hydroxide. In Expts. 30 to 42 of Table I, gold was added to the tissue just after it was put into the Kjeldahl flask. The results show that small amounts of gold can be determined quantitatively in tissue by our method. In Expt. 42, the only unsatisfactory case, a precipitate was present in the electrolyzed solu-

tion and could be seen embedded in the deposited gold. Of the other experiments, only 34 and 35 show an error of more than 0.04 mg., this being a maximum error of 4% on the basis of 2.5 mg. gold. When only 0.23 mg. is determined, however, the variation of 0.04 mg. means a possible 20% error. The usual source of error is incomplete washing of flasks or electrodes, but with the use of the assay balance, a weighing error of 0.02 to 0.03 mg. may occur unless extraordinary precautions are observed. Expts. 43 and 44 were run to decide the effect of the presence of precipitates as in Expt. 42. There was a precipitate in Expt. 44 and none in Expt. 43. The results indicate that the electrolyte must be free from precipitate if reliable results are to be obtained. Filtration through an alundum plate which was packed in place of asbestos gave satisfactory results in the removal of insoluble residue in the electrolyte. In a few cases after several gold solutions had been filtered through the same filter, the residue and plate were treated with aqua regia and the resulting solution electrolyzed, but no gold was ever found. In Expts. 45 to 50 the solutions contained less than 0.02 mg. gold, since we had found that this was about the amount of gold which we might expect to find in our tissues. Since the animals received their gold as cyanides, Analyses 51 to 52 were run on aurous cyanide and 53-54 on potassium aurocyanide. The results show that gold in the form of cyanide can be determined by the method under consideration.

In one experiment a given amount of gold was injected into a living guinea pig which was then killed and analyzed, *in toto*. Only about 50% of the gold was recovered but the residue which had been filtered out was purple and the electrolyzed solution was yellow, both presumably colored by gold. Apparently, therefore, the method is not adequate to recover all the gold when a large amount of tissue is used. Since it had already been shown that the gold can be completely recovered from a 10 g. sample of tissue, the amounts recovered from a 20 and a 10 g. sample of the same tissue were compared as follows:[1]

	Gold found in 20 g. Mg.	Gold found in 10 g. Mg.
Sample No. 1	22	16
Sample No. 2	23	17
Sample No. 3	14	13
Sample No. 4	10	11

It is evident that all the gold is not recovered from a 20 g. sample.

An 8-hole Kjeldahl stand was used in the digestion of the tissue and 6 electrolyses were run at once. The average time required for complete

[1] It was noted that the amount of mineral matter in samples of tissue containing bone, in feces, and in urine was great and that results of analyses of these samples were less dependable than those obtained from analyses of other tissues. 5 g. samples gave better results in these analyses unless the amounts of gold present were too small.

analysis of each of about 250 samples was less than two hours. The report of this analytical work has been published by DeWitt, Cadwell and Leavell.[1]

Final Procedure.

The procedure in detail as finally worked out after all the preliminary tests had been made was as follows: 10 g. samples of fresh tissue[2] are placed in 300 cc. Kjeldahl flasks; 10 cc. of C. P. conc. sulfuric acid and 10 cc. of C. P. conc. nitric acid are added to it. The mixture is digested over a gas flame with the addition of nitric acid as needed, until the cooled sulfuric acid solution is colorless. Glass beads are inserted to prevent bumping during digestion. A glass tube through which clean air can be forced is run part way down the neck of the flask. The flask and its contents are then heated while the white fumes are blown out until the solution is concentrated to about 2 cc. If the solution is dark, nitric acid must be added to it and the mixture heated until it is colorless. When the solution is colorless, the tube is withdrawn and one cc. each of C. P. conc. nitric and hydrochloric acids are added. The mixture is boiled a few minutes, then one cc. more of conc. hydrochloric acid is added and the solution boiled again.

After the solution is cooled and diluted with 5 cc. of distilled water, C. P. conc. ammonium hydroxide is added until the color is discharged, then an excess of 2 cc. After the solution has been boiled one minute, there should still be an odor of ammonia at the mouth of the flask. If such is not the case, add more ammonia and boil again. More or less white precipitate often appears at this point. After the mixture has cooled, 5 cc. of conc. hydrochloric acid are added and the solution is boiled three minutes. Most of the non-crystalline precipitate should dissolve, but the boiling should not be continued more than three minutes. If any gold is present, the solution is usually yellow at this point. This solution is filtered by suction through an alundum plate packed with well-washed asbestos into the beaker in which the electrolysis is to be carried out. The Kjeldahl flask is rinsed through the filter with water acidulated with hydrochloric acid.

Ammonium hydroxide is added to the filtrate until the odor of ammonia is distinct. The gold usually does not precipitate at this time, but hydroxides of other metals may come down. The solution is made faintly acid and warmed if necessary to dissolve all the precipitate. To the solution,

[1] *J. Pharmacol.*, 11, 357 (1918).

[2] If the tissue cannot be put directly into the Kjeldahl flask in which it is to be digested, it should be stored in small bottles and covered with 10 cc. of C. P. conc. sulfuric acid. The acid and undecomposed tissue can later be poured into a 300 cc. Kjeldahl flask. Any solid residue is stirred with C. P. conc. nitric acid and rinsed into the flask. The process is repeated with aqua regia to dissolve precipitated gold and then with water until the bottle is perfectly clean.

the volume of which should be about 40 cc., 1.1 cc. of 85% phosphoric acid and 0.75 g. of disodium hydrogen phosphate are added. It is then ready for electrolysis.

The electrodes are platinum. The cathode consists of a 1.5 cm. square plate of thin platinum foil to which is fused a platinum wire long enough to reach above the top of the beaker, and weighs less than 2 g. Any previous deposit of gold is first dissolved off with an approximately 3% solution of potassium cyanide and enough hydrogen peroxide to cause solution to take place quickly. Warming facilitates this reaction. After a thorough rinsing with water, nitric acid, water, alcohol, and ether, the electrode is dried and weighed.

During the electrolysis, the cathode is held below the level of the rotating anode by means of a bent glass rod. The temperature is kept at about 60°, but may vary from 50 to 70°. The electrode potential difference should never exceed 1.2 volts. The amperage may start as high as N. $D_{.100}$ = 0.07, but soon falls off considerably. After 40 minutes, the current is stopped and the cathode is withdrawn. It is very thoroughly washed with water then with alcohol and ether and dried at 140° for a few minutes, and weighed.

To demonstrate the correctness of this procedure, determinations were made of the percentage of the amount of gold injected into a living animal which could be recovered. Two guinea pigs were injected with a known amount of gold. They died a few hours later. Each body was ground, well mixed and duplicate 10 g. samples were analyzed. In one of the pigs 95% of the gold injected was accounted for and 100.6% in the other. In two other experiments, known amounts of gold were injected at intervals for two months into pigs which were kept in metabolism cages. By analyzing the excreta and samples of the bodies of the pigs, we were able to account for 92 and 97%, respectively, of the gold injected.

Determination of Larger Amounts of Gold in Inorganic Medium.—The above method having been proved satisfactory for the determination in organic materials of amounts of gold ranging from 2 mg. to 0.05 mg., it seemed desirable to test and, if necessary, modify the method so that it could be used for the determination of larger amounts of gold, in inorganic material, and for the separation of gold from iron and copper even when the latter are present in amounts nearly equal to that of gold.

This part of the investigation has been divided into 4 parts as follows: (1) the deposition of the gold as a bright adherent deposit from the fulminating gold solution; (2) the separation of gold from iron; (3) the separation of gold from copper; (4) the separation of gold from iron and copper.

The Precipitation of the Gold from Fulminating Gold Solution as a Bright Adherent Deposit.—As the solution of gold contained some free

chlorine, it was treated with ammonium hydroxide, etc., in the usual way.

In a preliminary experiment, 1.5 g. ammonium chloride was added to prevent the precipitation of gold. It was found that the deposition required 75 minutes. During the deposition there was some trouble with a precipitate and the final deposit was not sufficiently adherent. As this difficulty may have been due to the presence of too little ammonium chloride and too rapid precipitation of the gold, the experiments shown in Table II were run with varying amounts of ammonium chloride, a lower voltage and longer time. The voltage varied between 0.5 and 1.2, being kept at the lower figure for the first half hour; the current varied between 5 and 80 milliamperes. The time was 90 minutes.

TABLE II.

Deposition of Gold from Solutions with Varying Concentrations of Ammonium Chloride.

	Gold present. Mg.	NH_4Cl added. G.	Gold found. Mg.	Deposit.	Error. Mg.
1	31.5	3	31.4	Poor	0.1
2	31.5	3	31.1	Poor	0.4
3	31.5	6	31.2	Fair	0.3
4	31.5	6	31.2	Fair	0.3
5	31.5	9	31.4	Fair	0.1
6	31.5	9	31.6	Poor and solution spattered	0.1

The conditions of the third and fourth experiments seemed satisfactory and thereafter 6 g. of ammonium chloride was used in each electrolysis, and the voltage was kept at the lower value for at least the first thirty minutes.

The conclusions of the previous experiment were tested out in the series summarized in Table III. The voltage varied between 0.9 and 1.2 volts.

TABLE III.

Test Experiments on Deposition of Gold.

Gold present. Mg.	Gold found. Mg.	Error. Mg.	Remarks.
31.5	31.4	0.1	Good deposit
31.75	31.8	0.1	Good deposit
31.75	31.5	0.2	The deposit was not so good, as the rotating anode came too close to the cathode.

A final series summarized in Table IV was run in which it was the purpose to find the effect of varying the acidity. In this experiment the electrode potential difference was kept down to 0.5 volt for the first 50 minutes and at 1.1 volts for the last 40, making the total time for the deposition of the metal 90 minutes. The current varied from 0.35 to 0.02 ampere. In all cases the precipitates were good.

TABLE IV.

Deposition of Gold from Solutions with Varying Acidity.

Gold present. Mg.	Acidity.	Gold found. Mg.
31.75	slight as usual	31.6
31.75	slight as usual	31.6
31.75	2 cc. conc. hydrochloric in excess	31.6
31.75	2 cc. conc. hydrochloric in excess	31.65
31.75	large excess[1]	31.7
31.75	large excess[1]	31.8

The conclusion of these experiments is that gold can be determined satisfactorily from a fulminating gold solution according to the directions already given if to the solution of gold chloride containing from 0.03 to 0.04 g. of gold, 6. g of ammonium chloride is added to prevent precipitation, and the solution electrolyzed at 60° with a voltage of less than 0.6 volt for the first 30 to 45 minutes and below 1.3 for the remainder of the time. The deposition usually takes about 1.5 hours. It will be noted that the ammeter reading becomes very low at the end of the experiment, so can serve as an indication of the completion of the precipitation.

The Separation of Gold from Iron.—To determine whether gold could be deposited quantitatively in the presence of iron, one cc. of a ferric chloride solution containing about 0.025 g. of iron was added to the gold solution, and the experiment carried out as before. Throughout the experiment, the ammeter readings were greater, and the time for deposition was longer. Expts. 1 to 8 of Table V give the results.

The precipitates were dissolved off with potassium cyanide and hydrogen peroxide and were tested for iron with sodium thiocyanide, but no iron was found.

The Separation of Gold from Copper.—The experiments here were carried out just as in the case of the separation from iron, but one cc. of a solution of copper sulfate containing about 0.025 g. of copper was added to the solution of gold in each experiment. The results are shown in Table V, Expts. 9 to 13.

The deposits were all good. At the end of Expt. 10 the voltage became so high that a small amount of copper was deposited and this was removed by washing the electrode with dil. nitric acid before the final washing with water, alcohol, ether, and then drying.

The precipitates were dissolved off with potassium cyanide and hydrogen peroxide and tested for copper with ferrocyanide. No copper was present.

The Separation of Gold from Copper and Iron.—The experiments here were carried out just as in the cases of the copper and the iron separately,

[1] In these cases we first tried to electrolyze in an alkaline solution but the solution became cloudy and apparently conducted no current and several cc. of acid were added.

one cc. each of the iron and the copper solutions being added to each determination. The results are given in Table V, Expts. 14 to 18. The old deposits were all good.

TABLE V.

Deposition of Gold from Solutions Containing Iron and Copper.

	Gold present with iron. Mg.	Voltage. Volts.	Time. Hrs.	Gold found. Mg.	Error. Mg.
1	31.5[1]	0.6-1.2	2 hot	31.5	..
			14 cold		
2	31.75[2]	0.6-1.2	$3\frac{1}{2}$	31.8	..
3	31.75	0.6-1.2	$3\frac{1}{2}$	31.8	..
4	31.75	0.5-1.2	$2\frac{1}{4}$	31.7	..
5	31.75	0.5-1.2	$2\frac{1}{4}$	31.6	0.1
6	31.75	0.5-1.2	$2\frac{3}{4}$	31.6	0.1
7	31.75	0.6-1.2	2	31.8	0.05
8	31.75	0.6-1.2	2	31.7	0.05
9	31.5	0.5-1.2	2 hot	31.2	0.3
			10 cold		
10	31.5	0.5-1.2	same as (9)	31.3	0.2
11	31.75	0.5-1.2	$3\frac{1}{2}$	31.8	0.0
12	31.75	0.6-1.2	$3\frac{1}{2}$	31.7	0.0
13	31.75	0.6-1.2	$2\frac{1}{4}$	31.6	0.1
14	31.5[1]	0.7-1.1	$1\frac{1}{2}$ hot	31.3	0.2
			12 cold		
15	31.75	0.5-1.2	$3\frac{1}{2}$	31.7	0.0
16	31.75	0.5-1.2	$3\frac{1}{2}$	31.7	0.0
17	31.75	0.6-1.2	$2\frac{1}{4}$	31.7	0.0
18	31.75	0.6-1.2	$2\frac{1}{4}$	31.5	0.2

Conclusions.

1. With a maximum error of 0.05 mg., 3 mg. or less of gold, present as fulminating gold, can be completely deposited electrolytically in 40 minutes from a phosphoric acid solution at a temperature of 60°, using a rotating anode and an electrode difference of potential of from 0.9 to 1.2 volts.

2. By the addition of 6 g. of ammonium chloride to prevent precipitation, 30 to 40 mg. of gold can be electrolyzed at a temperature of 60°, with a voltage of less than 0.6 volt for the first 30 to 45 minutes and below 1.3 volts for the remainder of the time, complete deposition usually requiring about 1.5 hours.

3. Under the conditions stated in (1) and (2), gold can be completely separated from equivalent amounts of copper and iron, but the time required is greater than if no copper and iron are present.

4. It makes very little difference whether the electrolyzed solution is neutral or much more acid than prescribed.

5. Using the method described, it has been possible to recover in 4

[1] Anode not rotated while solution was cold.

[2] The voltage was kept low for the first hour.

consecutive cases more than 90% of the gold injected into living animals.

6. The average time required for carrying out a complete analysis for gold by this method has been less than two hours.

Credit is due to Mr. L. M. Larson for some preliminary work done on this problem.

We take this opportunity of expressing our gratitude to Dr. Lydia M. DeWitt, of the University of Chicago, for her helpful guidance.

CHICAGO, ILLINOIS.

[CONTRIBUTION FROM THE GEOPHYSICAL LABORATORY OF THE CARNEGIE INSTITUTION OF WASHINGTON.]

THE DETERMINATION OF THE COMPRESSIBILITY OF SOLIDS AT HIGH PRESSURES.

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The change of volume of a solid when subjected to hydrostatic pressure is a small quantity and one difficult to measure. It is perhaps for this reason that but few investigators have entered this field of experimentation. We have, to be sure, the measurements of Richards at comparatively low pressures, and those of Bridgman at much higher pressures, but for only three substances. Except for these measurements, however, there are practically no other data which represent the true compressibility of solids under hydrostatic pressure.

The original object of the authors in starting this investigation was to determine the compressibility of certain rocks and minerals, but preliminary experiments indicated that the method adopted was so satisfactory for solids in general that it seemed worth while to measure the compressibility at high pressures of some of the more common metals and other solids. Accordingly a number of such measurements have been made, and in what follows we describe the method we have used and present the results of our measurements on the compressibility at pressures up to 12,000 megabars of gold, copper, brass, silver, aluminum, zinc, tin, cadmium, lead, bismuth, a tin-bismuth alloy, sodium chloride, calcium-carbonate, and silica, both crystalline and amorphous.

General Description of Method.

The sample or test-piece of the material to be investigated is placed in the cylindrical bore of a thick-walled steel cylinder or bomb closed at the bottom and fitted at the top with a movable piston; and in order to transmit a uniform hydrostatic pressure to the test-piece the remainder of the space inside the bomb is filled with a thin liquid which will not solidify or thicken under pressure. If now pressure be applied by forcing the piston downward and simultaneous readings be taken of (1) the piston