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A comparison of the solvents used in the hydrometallurgy of silver

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Thesis.

A COMPARISON OF THE SOLVENTS USED IN THE
HYDROMETALLURGY OF SILVER.

Missouri School of Mines,
Rolla, Mo.

May 1909

Approved.

W. Copeland

Simon Schamberg

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8287

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A COMPARISON OF THE SOLVENTS USED IN THE HYDRO-
METALLURGY OF SILVER.

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Potassium cyanide solutions, hyposulphite of soda solutions, and a mixture called the Russell solution, are the solvents most used to win silver from its ores.

We are not the first to attempt comparisons of the solvent powers of the different solutions named. It is difficult, at least we have found it difficult, to find tabulated, a comparison of the various solvents from the points of view of speed of action, of consumption of reagents, of suitability of one or another to a given type of ore.

Such works as O. Hoffmann's most excellent book, "The Hydrometallurgy of Silver"; Carl A. Stetefeldt, Lixiviation of Silver Ores; H. F. Collins, Metallurgy of Silver; F. L. Bosqui, Notes on Cyanide Process; H. F. Julian & Smart, Cyaniding Gold and Silver Ores, together with articles appearing in the current journals, have been freely consulted. Wherever possible we have compared our results with those of other workers, and the assistance of Prof. D. Copeland has been a great help to us in all cases.

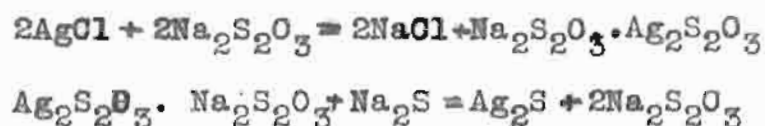
Outline of the Process.

Before going into the experiments in detail, we give an outline of the processes to which our work refers.

Hyposulphite Method.

This process is based on the solubility of silver and its compounds in dilute sodium hyposulphite solutions. The ore in vats, barrels or troughs is attacked by dilute hypo# solutions. The silver after solution is treated with some soluble sulphide, like sodium sulphide, which precipitates silver sulphide. The precipitate gives up its silver when roasted and smelted. The solution from the precipitation is used again as solvent on more ore.

Hypo supposedly acts best on those ores which naturally carry the silver as chloride or on ores which have been given a chloridizing roast. The following equation expresses the reaction:



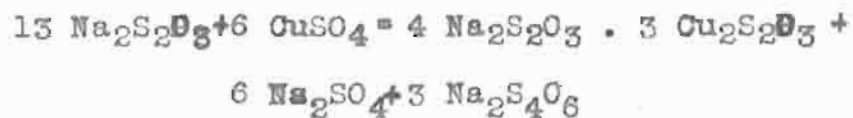
Russell Process

This method uses in addition to the ordinary hypo solution, a solution of copper sulphate with hypo.

Throughout this work "hypo" is used as an abbreviation of sodium hyposulphite.

The copper sulphate plus the hypo form a so-called double hyposulphite of copper and sodium which is known as "extra-solution". In ~~extra~~ solution silver sulphide is supposedly more soluble than in hypo solution alone. Solution and precipitation details are in general the same in this process as in the ordinary hypo method.

The chemical reaction is as follows:

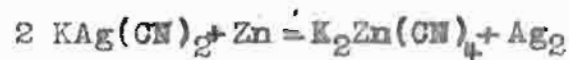
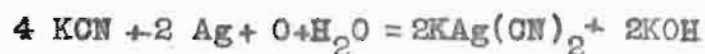


Potassium Cyanide Process.

Potassium cyanide for the last twenty years has played an important part in the metallurgy of gold. Of late, it is also being used as a solvent for silver. The process in general has the following steps: The ore, suitably prepared, is attacked with dilute potassium cyanide solutions, the resulting solution of silver filtered from the ore, and the silver precipitated from the solution by means of metallic zinc.

In this case, also the solution from the precipitate^{ion} is used as solvent on more ore.

The reactions are:



Ores.

Silver exists in ores in many conditions. Among the most

ordinary occurring silver minerals are argentite, (Silver sulphide, Ag_2S), Cerargyrite (Silver chloride, AgCl) and Pyrargyrite (Silver Sulphoantimonate, $3 \text{Ag}_2\text{S} \cdot \text{Sb}_2\text{S}_3$)

Problem.

1. If the silver exists in argentite, which of the three solutions, potassium cyanide, hypo, or Russell, is the best suited to dissolve the Silver?

2. If the silver exists as cerargyrite, which solution is best for dissolving the silver ?

3. Suppose silver exists as pyrargyrite, which solution will act the best ?

4. With each mineral and with each solution what is the loss of solvent in the process, what is the time required, and what is the best strength of solution?

These questions it was our purpose to solve.

Ore Used.

The silver in the ore used existed, pure as could be obtained, as argentite, cerargyrite and pyrargyrite. Enough of each was mixed with quartz (sand) to make the average 0.3% of silver (nearly 100 ounces per ton).

These mixtures served as our "ores". The ore contained in each mineral was treated under like conditions with each of the solvents.

1. Treatment of Argentite with hypo solutions.

Argentite is a silver sulphide. It contains, when pure, 87.1 percent of silver. Is insoluble in water, but soluble in potassium cyanide solutions. Its solubility in hypo solutions is somewhat doubtful.

Our ore was a mixture of argentite and quartz, containing 0.3 of one percent of silver, all crushed to 30 mesh. ^{Twenty grams of ore} ~~25~~ grams, containing 75 milligrams of silver was taken for each experiment. The ore was placed in a beaker and treated for 24 hours with the proper amount of solution. In practice the solution varies from four to five times the weight of ore treated, so 150 cubic centimeters of solution were used with the 25 grams of ore. During solution the ore was ^{not} ~~neither~~ agitated nor the solvent heated. The room temperature was 15 degrees C.

The cerargyrite and pyrargyrite were treated the same as was the argentite.

Solutions Used.

The hypo solutions used were of strength varying from 0.1% to 15 %. (0.1, 0.5, 1.1.5, 2, 3, 5, 10, 15%) Thus covering a much larger range than is found in practice.

After the ore was in contact with the solution for the time mentioned above, it was filtered and the residue after washing was dried and assayed for silver.

To the filtrate the precipitant, sodium sulphide was added. The sodium sulphide was prepared by dissolving caustic soda in hot water, and adding two-thirds of its weight of sulphur. After boiling, all the sulphur disappears, leaving a reddish yellow liquid, which was filtered. The sodium sulphide prepared in this way (the two-thirds rule) has, according to Russell, the formula $\text{Na}_2\text{S} + \text{Na}_2\text{S}_2$

A quantity of it was placed in a burette and added drop by drop to the hypo solution containing the silver. Precipitation follows almost immediately, giving a dark colored slow settling precipitate.

The addition of sodium sulphide was continued until no further precipitation took place.

The amount of sodium sulphide used was measured. The solution was filtered. The precipitate was assayed for silver by fusion, scorification and cupellation. The filtrate contained the remaining hypo and a slight excess of sodium sulphide. To determine the percent of free hypo still left in the filtrate, the following method was employed:

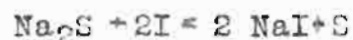
The quantity of solution was measured and analyzed for hypo. The total hypo in the solution added to the ore, less that in the solution after precipitation, gave the hypo consumption.

Then ten c.c. of ^{filtrate} ~~solvent~~ were titrated with iodine solution in presence of starch. The iodine solution was prepared, dissolving 5.11 grams of pure iodine per liter. Each cubic centimeter of the iodine solution is in these conditions equivalent to 0.1% of sodium hyposulphite, when 10 c.c. of the hypo solution are analyzed.

The starch solution was prepared boiling 10 grams of starch with 800 C.C. of water and then a strong solution of salt was added to prevent souring..

The sodium sulphide excess which had been used in precipitation, gave trouble during titration with the iodine.

Sodium sulphide with iodine reacts as follows:



There is thrown down the sulphur precipitate, with its characteristic yellowish-white color. The amount of iodine solution, therefore, indicated not only the hypo but also the sodium sulphide present. To obtain a correct estimation, the sodium sulphide had to be considered. ~~It was titrated separately against iodine.~~

The sulphur either remained as sodium sulphide or was precipitated as silver sulphide. The amount of silver was known ^{and}. The amount of sulphur with it, ^{was} calculated ~~and~~ the sodium sulphide ^{being known} used was ~~measured~~. The sodium sulphide remaining in solution, reacting with the iodine, was calculated and connection was made for this ~~sodium sulphide~~ in reporting the hypo results.

The analysis of the sodium sulphide was made by two ways, that gave close checks.

1. By adding an excess of copper sulphate, obtaining cupric sulphide and sodium sulphate.



The cupric sulphide was filtered, dried and weighed, and its contents in sulphur calculated, corresponding ^{closely} to the amount of sodium sulphide taken. This method is not ~~exact~~ as the sodium sulphide contains some polysulphides that will give the same reaction, with a liberation of free sulphur.



2. By oxidation with nitric acid. All the sulphur was changed to sulphuric acid, and then weighed as barium sulphate.

Results.

The results obtained from the treatment of Argentite by hypo solutions are shown in Table No. I and graphically in Plate No. I.

We note that there is a rapid increase in solubility of silver as the solutions grow stronger. Solutions varying from one to three percent of hypo have approximately the same solvent power. This suggests that no advantage is to be gained by increasing the hypo solution from one to three percent strength.

As to the hypo consumption, we note that with solutions containing from 5% hypo up, there is a stronger solution left after precipitation, the other ^{strength} remaining unchanged, this being the most desirable case. A solution that gets stronger all the time is hard to keep at the required strength, and in this process becomes a solvent not only for the minerals we are after but also for others we do not desire in solution, bringing impurities and worthless material with all its consequences.

The plates are designed as follows:

The full lines show the silver extraction with different strengths of hypo. The dotted lines indicate the percent of hypo remaining in solution after using. The difference between the two curves is in each case the hypo consumption. It will be observed that the two curves are

somewhat similar.

TREATMENT OF ARGENTITE WITH RUSSELL SOLUTIONS.

The experiments described for argentite with hypo solutions were repeated with Russell solutions. The same strength of the solution was used in hypo (0.1% 0.5, 1, 1.5, 2, 3, 5, 10, 15%) Adding to them copper sulphate in from one half to one third the weight of hypo. The solutions used had then the following percentage.

Sodium Hypo.	Copper Sulphate.
%	%
0.1	0.06
0.5	0.2
1.0	0.4
1.5	0.6
2.	0.8
3.	1.0
5	2.0
10.	3.0
15	6.0

The ore was attacked in the same way as with hypo (150 c.c. at the room temperature, with 25 grams of ore, for 24 hours). The precipitation required a larger amount of sodium sulphide, owing to the necessity of precipitating the copper from the solution at the same time ^{as} ~~than~~ the silver. When assaying, careful scorification had to be done, on account of the presence of copper.

All the other details were the same as for hypo. The results obtained are shown in Table II and Plate I. In Plate I we note that in contrast with the hypo solution, the Russell solution gives a greatly increased extraction for very small increase in solution strength. This is true of Russell solutions only up to 5% hypo. After passing 5%, further increases of strength of Russell solutions gave no added silver extraction. (We classify the Russell solutions according to the percentage of hypo they contain.) The recovery of hypo from Russell solutions is small. With a solution containing before using 1% ^{sodium} ~~potassium~~ ^{hypo} cyanide, the consumption of hypo during the process was complete. A table showing the percentage of extraction of silver gained by using Russell solutions instead of hypo solutions is given the number X.

Treatment of Argentite with Potassium Cyanide Solutions.

The solutions used were of six different strengths, ~~0.1%, 0.2, 0.4, 0.6, 0.8, and 1%.~~ 150 c.c. were also used on 25 grams of ore. The tailings were assayed for silver, and the solution left titrated for free potassium cyanide with silver nitrate. The silver nitrate solution was made ^{by} dissolving 13.07 grams of that salt in one liter of water, in which condition 1 c.c. of it is equivalent to 0.1% of potassium cyanide when 10 c.c. of the cyanide solution are taken for titration.

The results obtained in these experiments are shown in Table III and in Plate II.

Increasing the solution strength from 0.1% to 0.2% potassium cyanide, causes a rapid increase in silver extraction, but beyond 0.2% potassium cyanide a farther increase in solution strength gives no corresponding increase in extraction. With solutions up to 0.2% the consumption of KCN is complete. This shows us the reason why solutions weaker than 0.2% gave a lower extraction of silver, and also points to the statement that provided there be unconsumed potassium cyanide present, added strength of solution is of no benefit when treating argentite ores. All the potassium cyanide in a solution as strong as 0.2% seems to have been used up in 24 hours. In treating an ore under our conditions it would be necessary to provide KCN in sufficient amount to satisfy this rate of consumption. The rate of silver solution, and therefore the rate of potassium cyanide consumption should vary with the amount of surface of silver mineral exposed and hence with the fineness of crushing.

Comparison of Hypo, Russell and Cyanide Solutions as solvents for Argentite.

The greatest extraction of silver is given by potassium cyanide solutions, ^{giving extraction as high} that goes as far as 45% (for our experiments) whereas the Russell solution gave a maximum of 29.33 and the hypo only 21.28.

In case an ore containing large amounts of argentite had to be leached raw, potassium cyanide would be the best solvent. When leaching with this solution, the strength is unimportant, always provided there be present some free KCN after precipitation.

If for some reason, Russell solution had to be used, the best strength would be between three and five percent, but never greater than that. Hypo solutions are not suited to raw argentite ores.

The above statements are our answers to the first question of the problem: if the silver exists as argentite, which of the three solutions, potassium cyanide, hypo, or Russell, is best suited to dissolve the silver ?.

Treatment of Cerargyrite with Hypo Solution.

Cerargyrite contains 75.3% silver. Is an important silver ore.

The method used was the same as explained in the preceding experiments.

The results are shown in Table IV. and Plate III.

In spite of the well known solubility of silver chloride (cerargyrite) in hypo solutions, our experiments do not show it in the best shape. The increase in solubility is almost uniform as solutions grow stronger, excepting between three and five percent, where remains the same. The hypo recovery is good up to 10%, from which point the recovery decreases.

Treatment of Cerargyrite with Russell Solutions.

Table V. and Plate III ~~give a good idea of~~ ^{show} the results obtained.

A solution of 3% seems to be the most desirable, as the extraction of silver decreases as the solutions increase to 5%. From 5% on to 10%, increasing the strength gives added silver extraction. Beyond 10% hypo, the extraction again falls down.

The hypo consumption is small for solutions between 1 and 3%. In general the hypo left in solution increases and decreases in the same way as the silver extraction. A good silver extraction therefore is accompanied by small consumption of hypo, while poor silver extraction is accompanied by big consumption.

Treatment of Cerargyrite with Potassium Cyanide Solutions.

The work is better shown in Table VI. and Plate IV. The silver extraction increases with an increased strength of potassium cyanide solution. At least, this is true for ^{or} ~~one~~ range of solution strengths.

Solutions up to 0.1% show a complete consumption of KCN. The actual potassium cyanide consumed is greater the stronger the solution used. Under our conditions we can point to no best strength of solution. All that we can say is that the stronger the solution, the better the silver extraction, giving also a greater KCN consumption.

These conditions might be changed with finer crushing.

Comparison between hypo, Russell and Cyanide for Treatment of Cerargyrite.

In case an ore containing silver as cerargyrite had to be leached, potassium cyanide would be the most effective solvent.

Of our minerals, the cerargyrite was the one of which we had doubts as to its purity.

The above statements answer only imperfectly the second part of our problem.

Treatment of Pyrargyrite with Hypo Solution.

Refer to Table VII and Plate V.

We note: that the hypo must be stronger than 2% if it is to rapidly attack pyrargyrite, solutions weaker than that are slow acting. With solutions at the start below 3%, there is in the process a hypo consumption, with solutions above 3% there is ^a hypo increase during the process.

Treatment of Pyrargyrite with Russell Solution.

See Table VIII and Plate V.

Pyrargyrite is one of the few common silver minerals that is less soluble in Russell solutions than in hypo solutions. This only applies for solutions that contain less than 2.2% of hypo, as for weaker solutions the extraction of Silver is greater with the Russell solution. The recovery of hypo is, as for most of the Russell cases, small.

Treatment of Pyrargyrite with Potassium Cyanide Solutions.

In plate II and Table IX, our results are shown. The solubility of pyrargyrite in this solvent, much resembles that of argentite, that is, a rapid increase for solutions varying from 0.1 to 0.2% KCN in strength. From there up, a farther increase in solution strength does not give a corresponding increase in silver extraction. With a solvent at the start as strong as about 0.2%, the consumption of potassium cyanide is complete. We wish to refer to the reasoning on the rate of consumption of KCN on argentite. The same reasoning applies here.

Comparison between hypo, Russell, and Cyanide Solution
as Solvent of Pyrargyrite.

In case an ore containing silver as pyrargyrite had to be leached raw, potassium cyanide is the best solvent. The strength of KCN solution used is unimportant, provided it be at least 0.2% and that there be always free KCN in solution.

The preceding statements answer the third part of our problem.

As to the fourth part, we have answered it for each case.

Table I.

Showing the solubility in hypo of the silver existing as Argentite; and hypo consumption

Strength of Hypo solution	Percent of silver extraction	Strength in hypo of solution after precipitation
$\frac{\%}{100}$	$\%$	$\frac{\%}{100}$
0.1	0.133	0.10
0.5	0.173	0.50
1.0	0.427	1.00
1.5	0.507	1.50
2.0	0.540	2.00
3.0	0.933	3.00
5.0	2.172	5.00
10.0	9.773	10.80
15.0	21.280	15.87

TABLE II.

of the Silver

Showing the Solubility in Russel) Solutions, Existing
as Argentite and ^{the} Hypo consumption.

Strength of solution %		Percent of Extraction <small>by lot</small> %	Percent of Free hypo after precip.
Hypo	CuSO ₄		
0.1	0.06	0.187	0.00
0.5	0.20	10.973	0.00
1.0	0.40	13.293	0.00
1.5	0.60	14.680	0.50
2.0	0.80	18.653	0.70
3.0	1.00	23.546	0.96
5.0	2.00	29.200	2.70
10.0	3.00	29.333	3.98

TABLE III.

Solubility of the Silver Contained in Argentite in Potassium Cyanide Solutions and Consumption of Solvent.

Strength of solutions %	Silver extraction %	Strength of Solutions after precipitation. %
0.1	37.86	0.005
0.2	40.27	0.010
0.4	40.80	0.190
0.6	41.47	0.22
0.8	42.00	0.32
1.0	45.00	0.87

TABLE IV.

Showing the Solubility of the Silver Contained in
Argentite in hypo solutions and hypo consumption.

Strength of solution %	Extraction of Ag. $\frac{1}{2}$	Strength of solution after precip %
0.1	0.426	0.1
0.5	0.386	0.5
1.0	1.466	1.0
1.5	1.600	1.5
2.0	2.013	2.0
3.0	3.080	3.0
5.0	3.333	5.0
10.0	6.453	10.0
15.0	9.000	12.4

TABLE V.

Solubility of Silver Contained in Cerargyrite in Russel
Solutions, and Hypo Consumption.

Strength of Solution		Extraction	Percent of
%	%	of Silver	free hypo
Hypo	Copper Sulphate	%	after precip.
0.1	0.06	0.106	0.00
0.5	0.20	0.586	0.00
1.0	0.40	2.640	0.00
1.5	0.60	4.080	1.02
2.0	0.80	13.066	0.84
3.0	1.00	20.400	1.84
5.0	2.00	13.333	1.92
10.0	3.00	22.146	5.20
15.0	5.00	16.933	9.31

TABLE VI.

Solubility in Potassium Cyanide Solution of the Silver
 Contained in Cerargyrite and Consumption of Solvent.

Strength of solution % KCN	Percent of extraction of silver	Strength of solution after precip. % KCN
0.1	19.866	0.01
0.2	32.000	0.038
0.4	44.00	0.072
0.6	49.01	0.12
0.8	73.36	0.18
1.0	78.94	0.25

Table VII.

Solubility of the Silver Contained in Pyrargyrite in hypo solution, and Hypo Consumption

Strength of solution	Extraction of silver	Strength of solution after precip.
% Hypo	%	% Hypo
0.1	0.000	0.10
0.5	0.000	0.50
1.0	0.000	1.46
1.5	0.066	1.47
2.0	0.372	1.74
3.0	5.628	2.95
5.0	7.220	5.55
10.0	21.133	12.00
15.0	27.640	17.03

TABLE VIII.

Solubility of the Silver contained in Pyrargyrite
with Russel Solutions.

Strength of Solution		Percent of Extraction	Percent of free hypo after precip.
Hypo %	CuSO ₄ %		
0.1	0.06	0.000	0.00
0.5	0.20	0.600	0.00
1.0	0.40	0.586	0.56
1.5	0.60	0.693	0.84
2.0	0.80	0.880	0.57
3.0	1.00	0.733	0.51
5.0	2.00	3.466	1.26
10.0	3.00	5.733	2.10
15.0	5.00	8.746	5.58

TABLE IX.

Solubility of the Silver contained in Pyrrargyrite
in Potassium Cyanide solutions, and Consumption of Solvent.

Strength of solution	Percent of silver extraction	Strength of Solution after Precip.
% KCN	%	% KCW
0.1	19.68	0.02
0.2	25.16	0.03
0.4	25.76	0.11
0.6	27.06	0.28
0.8	28.82	0.43
1.0	30.12	0.75

TABLE X.

Table showing the difference in extraction of Silver from Argentite and Cerargyrite with hypo and Russel Solutions,

Strength of Solution in hypo Percent of Extraction Increased with Russel Solution.

<u>%</u>	<u>Argentite</u>	<u>Cerargyrite</u>
0.1	0.054	0.320
0.5	10.300	0.200
1.0	12.800	1.174
1.5	14.173	2.480
2.0	18.113	11.053
3.0	22.613	17.320
5.0	27.028	10.000
10.0	19.560	15.693
15.0		7.933

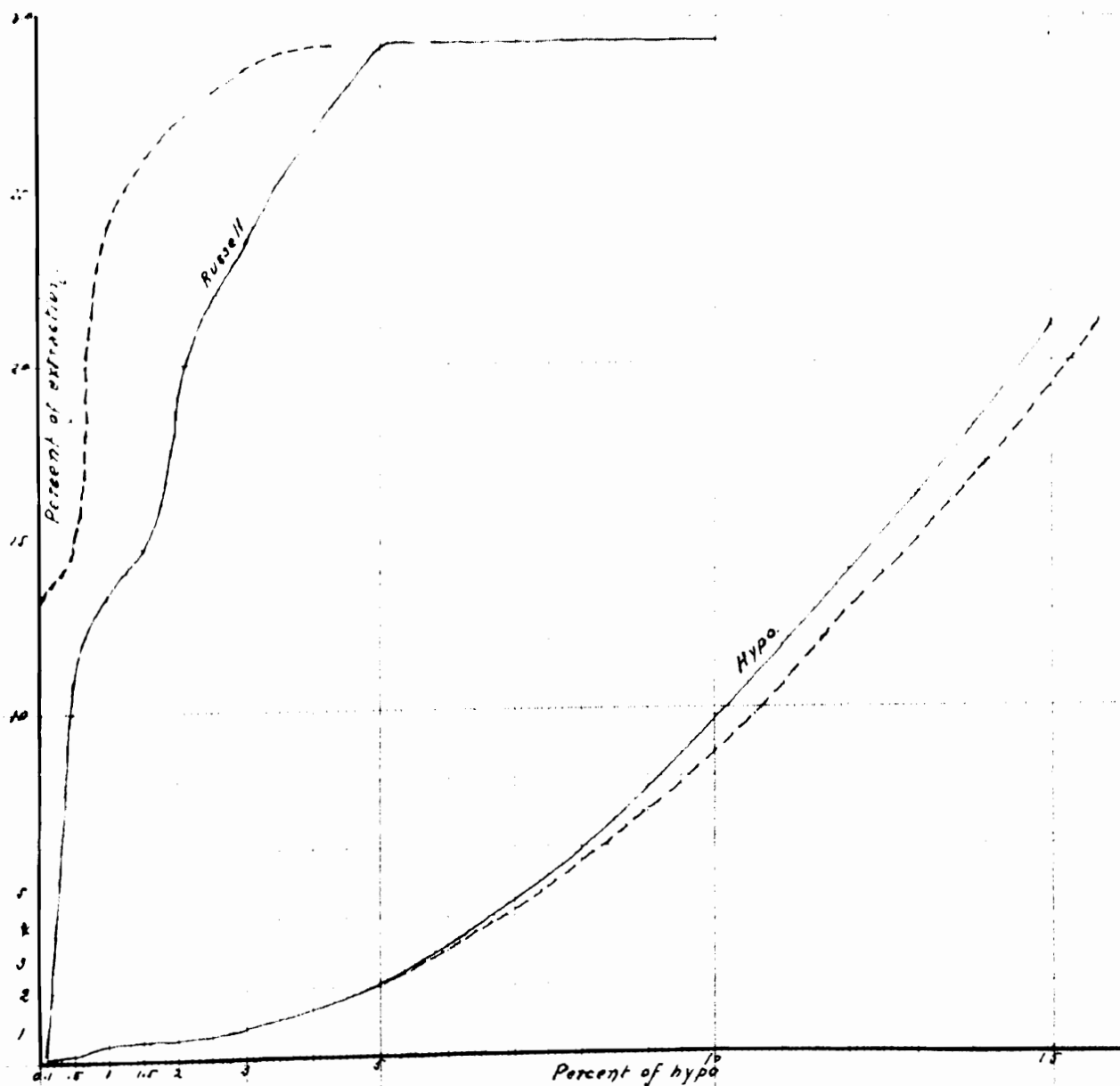
TABLE XI.

Table Showing the Difference in Extraction of Silver from Pyrrargyrite with hypo and Russel Solutions.

Strength of solution in hypo %	Difference in Percent of extraction		Diff. in %.
	Hypo (increased)	Russel (decreased)	of remaining hypo Russel (decreased)
0.1	0.000	----	0.10
0.5	0.000	----	0.50
1.0	0.586	----	0.90
1.5	0.627	----	0.63
2.0	0.508	----	1.17
3.0	----	4.853	2.44
5.0	----	3.754	4.39
10.0	----	15.400	9.90
15.0	----	18.894	11.45

PLATE I

Showing comparative extraction of Silver from Argentite with Hypo and Russol Solutions, and Hypo Consumption.



Scales
 % Hypo. 0 5 10 15 20
 % Extract. 0 1 2 3 4

PLATE II.

Showing the Solubility in Potassium Cyanide solutions, and Cyanide Consumption, of the Silver from Argentite and Pyrargyrite.

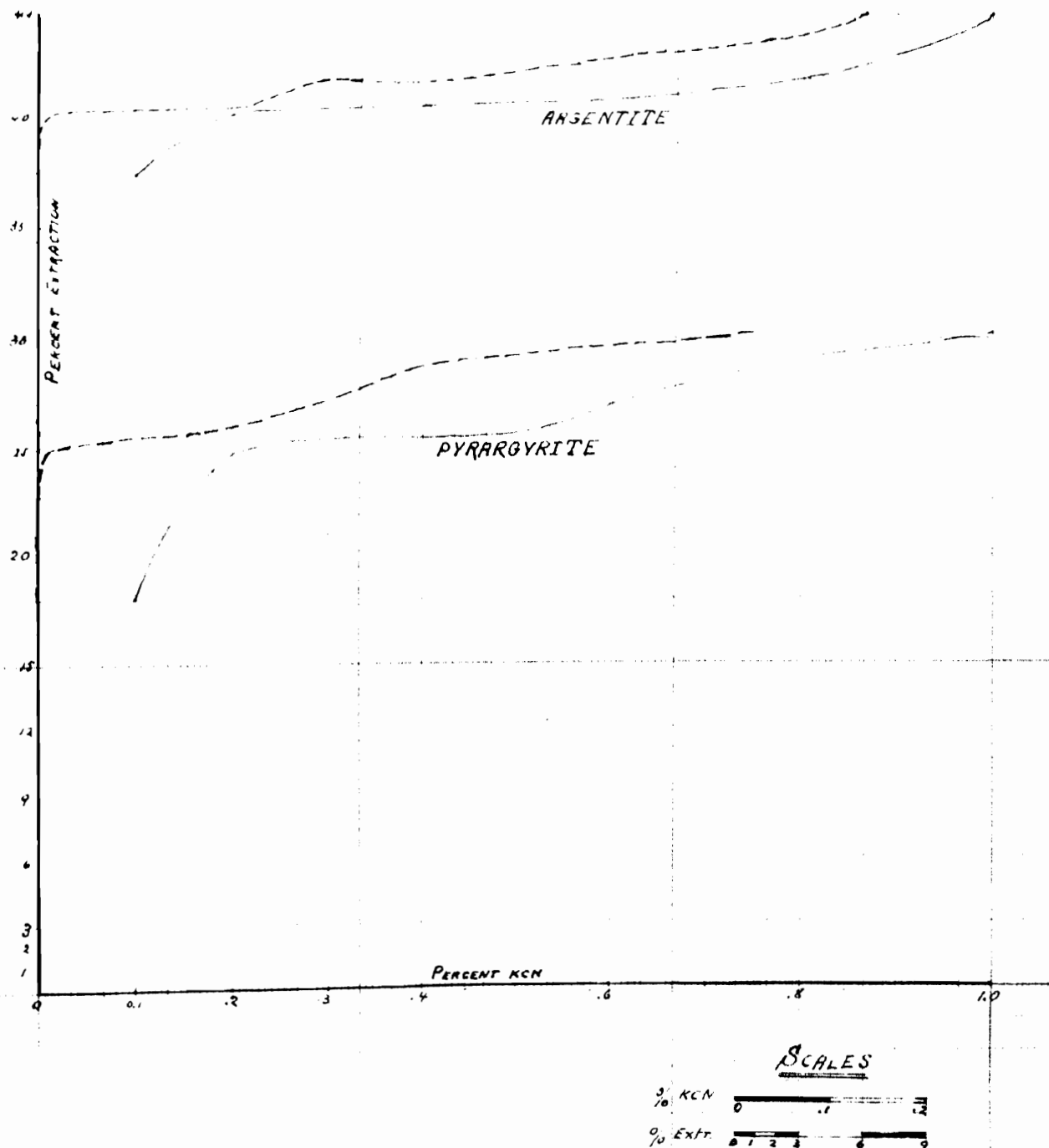


PLATE III.

Showing Solubility of the Silver from Cerargyrite in Hypo and Russel solutions, and Hypo Consumption.

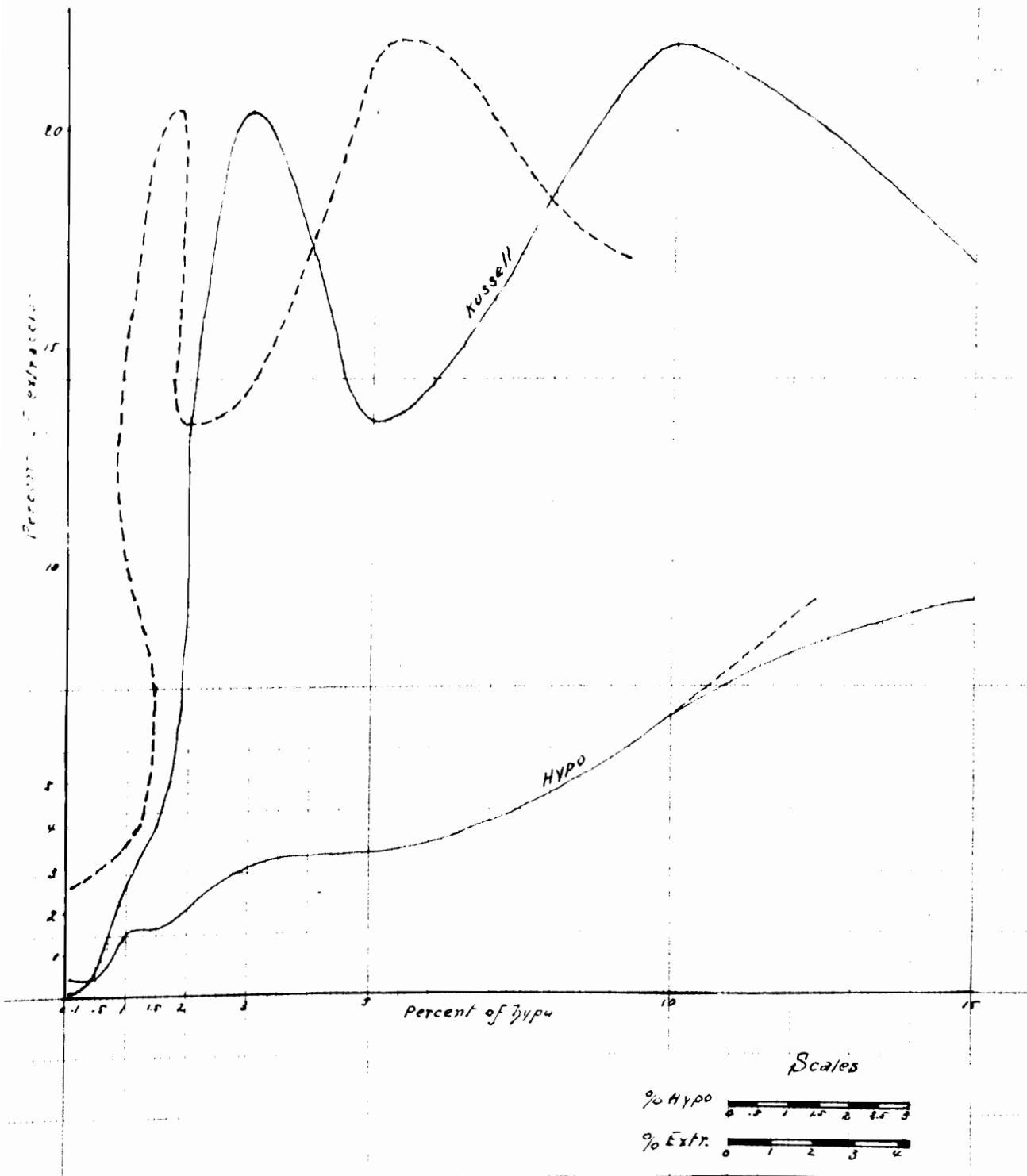


PLATE IV.

Showing the solubility of the Silver from Cerargyrite
in Potassium Cyanide solutions, and Solvent Consumption

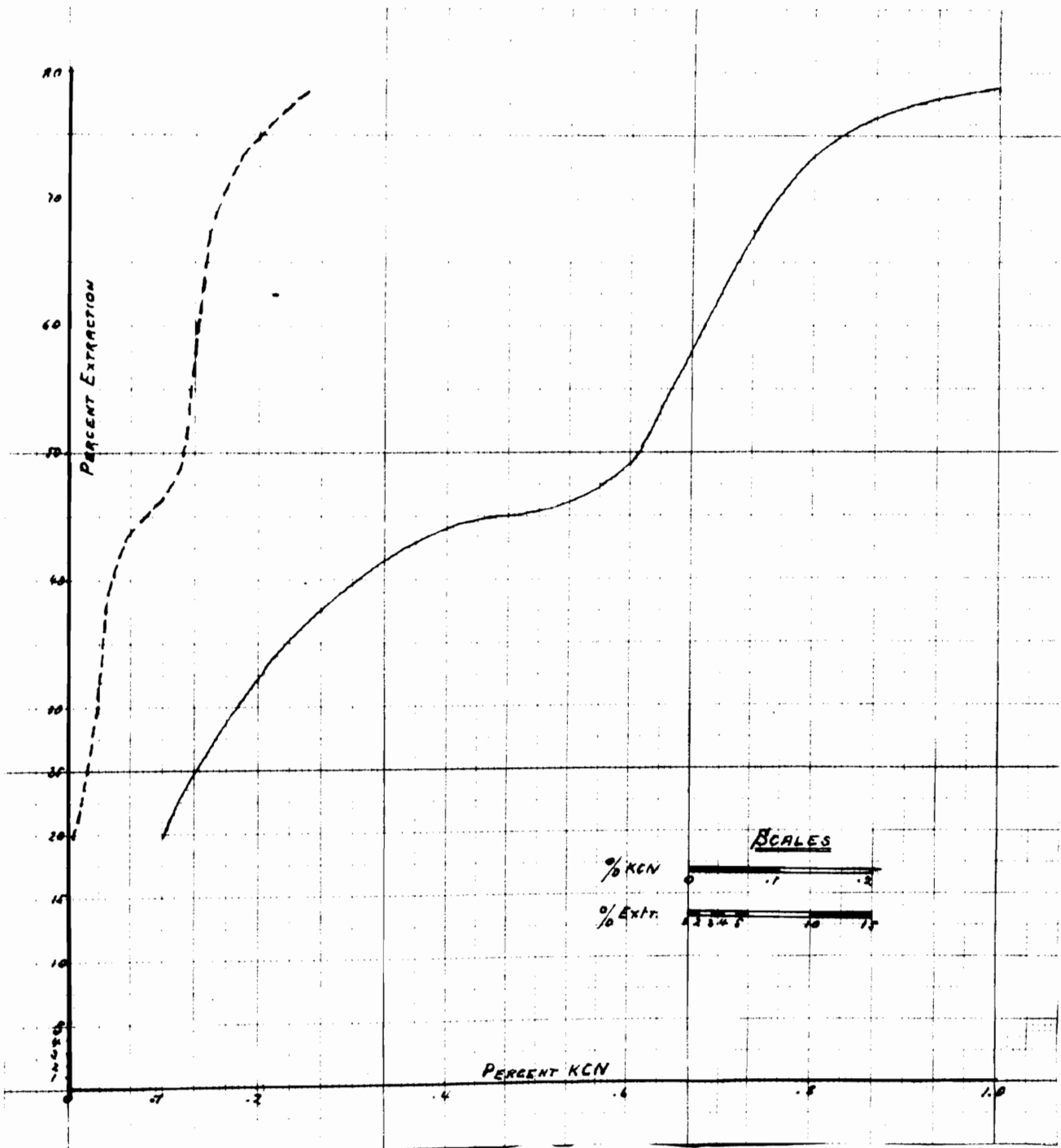
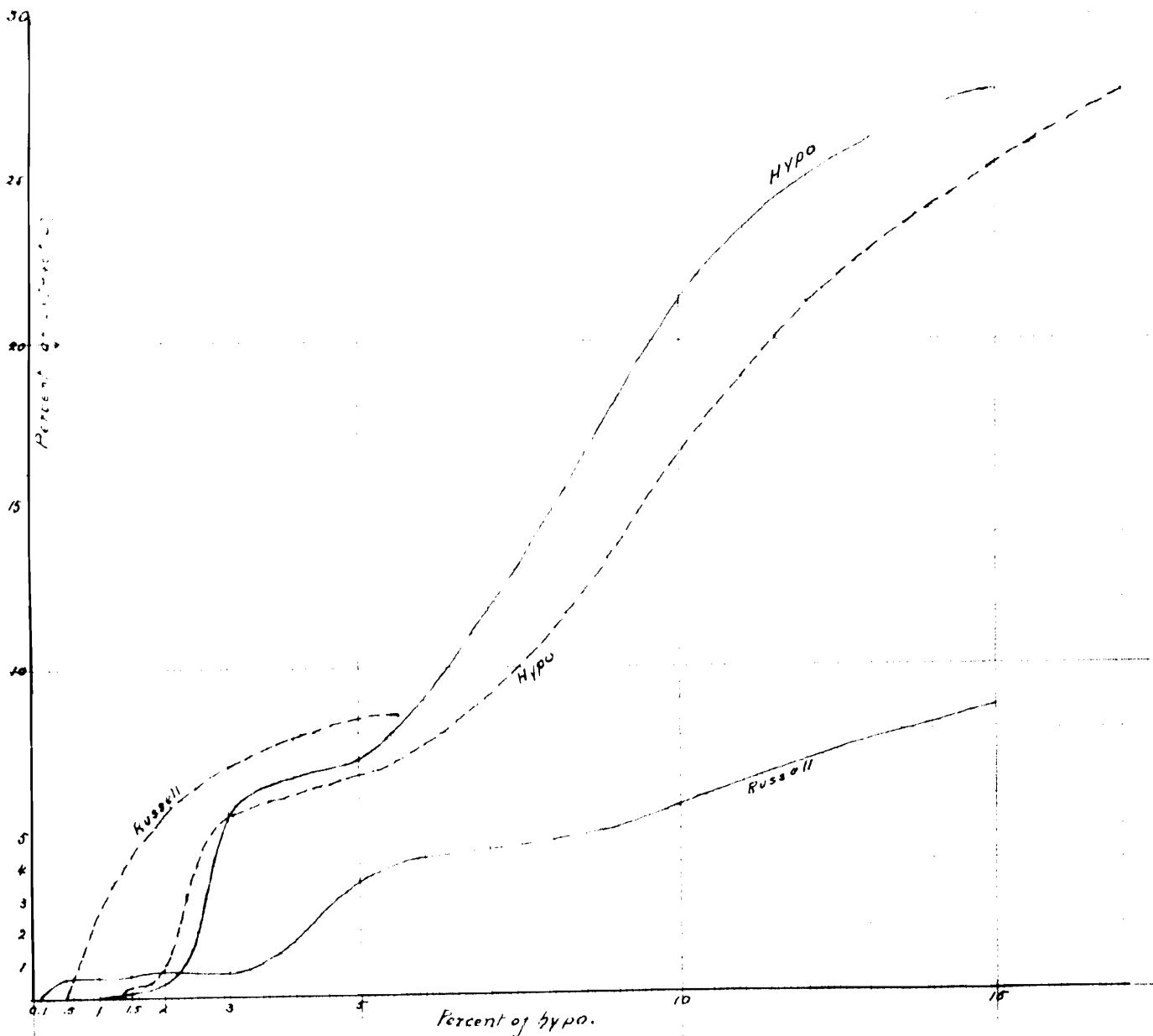


PLATE V.

Showing Solubility in Hypo solutions of the Silver contained in Pyrrargyrite and Hypo Consumption.



SCALES.

