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DISTRIBUTION OF My IN NITRIC ACID—DIGESTIONS
OF PITCHBLENDE—QUALITATIVE NOTES ON
FILTERABILITY OF GANGUES.

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by

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Progress Report

Radon
Distribution of My in Nitric Acid Digestions of Pitchblende
Qualitative Notes on Filterability of Gangues

by

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Three groups of experiments have been carried out with the purpose of determining the distribution of My in the nitric acid digestions of rich pitchblende. Incidental to this work some qualitative information has been obtained on the character of the gangues resulting from the digestions. The My assays on these experiments are not complete, and few significant conclusions can be made as to My distribution at this time. The completed experiments will be reported at a future date.

The various experiments were designed to determine:

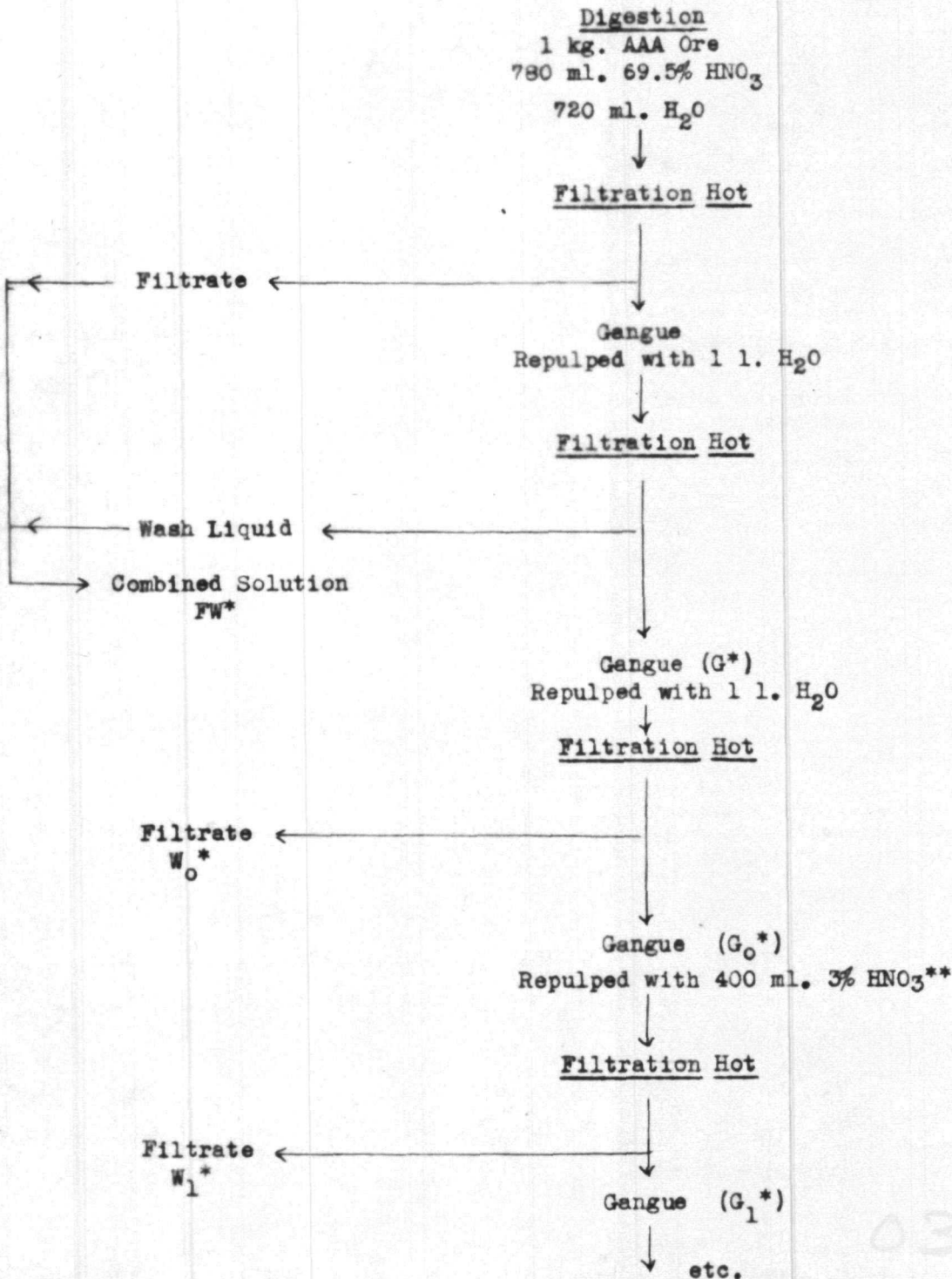
- I The effect of washing on the removal of My from the gangue.
- II The effect of excess nitric acid and time of digestion on the removal of My from the ore.
- III The effect of the concentration of nitric acid on the extraction of My.

I The Effect of Washing

In Fig. 1 is given a flow sheet of a digestion experiment on kilogram portions of AAA ore. This experiment was carried out as nearly as possible in duplicate. However, it is to be noted that the handling of the large gelatinous gangues and the large volumes of solution cannot be carried out in a strictly quantitative manner. The sampling for My assay in the two experiments was identical except that in the second experiment one additional gangue sample was taken after the first repulping.

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Figure 1.
Flow Sheet for Repulp Washing
(AAA Ore through 100 mesh)



**Repeated for W₂*, W₃*, W₄*, W₅*; G₂*, G₃*, G₄*, G₅*.

*Denotes samples taken for My analysis.

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Table I

Sample No.	Digestion 1	Digestion 2
	% of Input My	% of Input My
FW	28.7%	43.7%
W ₀	2.1	3.8
W ₁	0.6	0.9
W ₂	0.5	0.6
W ₃	0.2	0.5
W ₄	0.1	0.6
W ₅	0.3	1.4
G ₅	<u>25.0</u>	<u>26.4</u>
Total recovery	57.5%	77.9%
	Weight My*	Weight My*
G	-	3.1×10^{-5} g.
G ₀	4.3×10^{-5} g.	6.9
G ₁	2.7	3.6
G ₂	6.2	4.3
G ₃	5.7	-
G ₄	7.3	3.7
G ₅	5.1	5.4

*These values do not figure in the material balance. Their scattering probably reflects the difficulty in sampling.

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II The Effect of Excess HNO_3 and Time of Digestion on Removal of
 My from the Ore

This group of experiments consisted of 8 digestions, performed in duplicate, wherein the stoichiometric proportions of HNO_3 and the times of digestion were varied. The main purpose of these experiments was the determination of the Pb, 308, and My residues in the gangue after the digestions.

TABLE II-A
 Ore through 100 mesh.

Exp. No.	Time Di- gestion, Hr.	Stoich. HNO_3	Conc. HNO_3	Wash method
1A	1	2x	36%	Direct
1B	1	2x	36%	Repulp
2A	2	2x	36%	Direct
2B	2	2x	36%	Repulp
3A	3	2x	36%	Direct
3B	3	2x	36%	Repulp
5A	1	3x	54%	Direct
5B	1	3x	54%	Repulp
6A	2	3x	54%	Direct
6B	2	3x	54%	Repulp
7A	3	3x	54%	Direct
7B	3	3x	54%	Repulp
9A	1	4x	69.5%	Direct
9B	1	4x	69.5%	Repulp
10A	2	4x	69.5%	Direct
10B	2	4x	69.5%	Repulp

Acid mixture volume was kept as near 150 ml. as possible, to approximate large scale (30 kilo.) digestions being carried out by others in this Laboratory.

In each case 100 g. of ore was placed in a 500 ml. round-bottomed flask, and the acid mixture added slowly. The rate of acid addition was dependent on the violence of the reaction. When the 4x experiments were run, the reaction flasks had to be cooled with a bath of ice to keep the reaction under control. After the initial reaction subsided, the flasks were placed in a water bath, kept at 85-95°, and stirred vigorously during the requisite digestion period.

It seems apparent that the 308 goes into solution very soon after the addition of the acid. Whether long digestion times aid the solution of Pb and My from the ore is still uncertain.

Filtration Details.

After the completion of the digestion the hot slurry was filtered on a Buchner funnel, using "varnitized" cloth as the filter medium. The differential pressure between the atmosphere and the system was kept as near 60 cm. Hg as possible.

The characteristics of the gangues did not vary greatly, but the 4x gangues did seem more granular than the others.

The filter medium, "varnitized" cloth, was suitable in most cases. However, the slurries from the 4x digestions had to be diluted with 100 ml. hot water to prevent the cloth from suffering extensive damage.

The "A" experiments of Table II-A were washed by direct displacement through the cake after all the free filtrate had been drawn through. 250 ml. of hot 3% HNO₃ was used as the wash liquid. In the "B" series of Table II^A the gangue was repulped with 200 ml. of hot 3% HNO for 15 minutes. The pulp was transferred to the filter with 50 ml. 3% HNO₃.

No quantitative filtering data were obtained in these experiments; however, the following observations were made: (1) The more concentrated acid solutions gave the more easily filterable gangue. (2) After repulping filtration was more rapid than in the direct washing procedure. This latter result is, of course, to be expected.

Table II-B gives the results of the examinations of the gangues which have been obtained to date.

Table II-B
308 Recovery in each case > 99%.

Experiment No.	Percent of Input PbO in Gangue
1A	< 0.1%
1B	< 0.1
2A	3.5
2B	4.0
3A	4.0
3B	5.5
5A	< 0.1
5B	8.5*
6A	< 0.1
6B	< 0.1
7A	< 0.1
7B	2.3
9A	< 0.1
9B	< 0.1
10A	< 0.1
10B	< 0.1

*Probably due to faulty washing.

The lead determinations were made by the standard sulfate-chromate method after the gangues had been attacked by HF and H₂SO₄. The filtrates from the lead sulfate were compared in color, by eye, with a similar solution containing, as the sulfate, that weight of 308 corresponding to a 1% loss in the gangue. This procedure was checked by direct analysis as will be described below.

It is immediately apparent that the repulp procedure gave the less effective washing of the gangue. In every case essentially complete recovery of 308 was obtained. Those cases in which lead was incompletely removed may well have been due to the washing procedure adopted. This procedure was an attempt to make these small scale experiments nearly correspond to the 30 kilo digestions being carried out

by others in this Laboratory.

Since the recovery of 308 was so nearly complete in all of the above described experiments, it was thought worth while to carry out experiments designed to determine the minimum excess of nitric acid at which complete extraction could be obtained. Table III-A gives the conditions of the second series of digestions at small excess of acid.

Table III-A
Ore through 100 mesh. Digestion time 1 hour.

Experiment No.	Stoich. HNO ₃	Conc. HNO ₃	Wash Method
1dA	1.25	23%	Direct
1dB	1.25	23	Repulp
2dA	1.50	27	Direct
2dB	1.50	27	Repulp
3dA	1.75	32	Direct
3dB	1.75	32	Repulp

Table III-B gives the results of the gangue examinations. In order to confirm the semi-quantitative determination of 308 in the gangues, the gangue from the experiment using 1.25x nitric acid and direct washing on the filter was carefully assayed by the cupferron - silver reductor method. The result showed 99.8% extraction.

Table III-B
308 Recovery in each case > 99%.

Experiment No.	Percent of Input PbO in Gangue
1dA*	12.0%
1dB	14.0
2dA	11.0
2dB	14.5
3dA	3.0
3dB	7.0

*Gangue assayed for 308; see text.

III The Effect of the Concentration of Nitric Acid on the
Extraction of My

The My analyses on the experiments now to be described are as yet incomplete. It is, however, possible to give the results obtained on the filterability of the gangues from the digestions. These experiments were carried out on kilogram portions of undried AAA ore (assay 67.3% 308). The nitric acid in appropriate excess and concentration was added directly to the ore. The digestion was carried out with efficient stirring for either three or five hour periods, the container being immersed in a water bath maintained at 85-95°. On removal from the water bath the digestion mixture was allowed to stand for three hours. This step was introduced as an approximation to conditions which might obtain in large scale work. The mixture was then agitated and filtered by suction on paper supported in a Buchner funnel by a layer of 1/8 inch filter cloth. Differential pressure was maintained roughly at 60 cm. Hg. After the free filtrate had passed through the filter, one liter of 3% nitric acid was poured slowly over the cake to displace most of the dissolved material. The cake was removed as well as possible from the filter, weighed, dried, reweighed and pulverized for My assay. The total volume of the filtrate, including the wash solution, was noted as was the time of filtration. On the ten experiments for which filtration proceeded properly, data were obtained sufficient to calculate the filtration rates.

Table IV gives the details of the various digestions and the results on filtering rates. The filter medium for the 10% digestions was inadequate; the fine slurry failed to stop on the paper. The one result obtained for this case should be given little weight. The most significant results of this work are apparent when one examines the rates for the 20% digestion as compared with the rates for the 30% and 40% digestions. The gangue from the 20% digestion was of a gelatinous nature and was extremely difficult to filter and wash. It is thought that filtration will give a minimum of difficulty for digestions carried out in more concentrated acids. If, however, dilute acids should be desirable for other reasons, further work on filterability

in the low ranges of HNO_3 concentration will be necessary.

The examinations of the dried gangues for their My content are now being carried out. The results, which these experiments were designed to give, will be reported later.

Table IV
2x Digestions (actual HNO_3 used was 2.08 stoichiometric)

Experiment No.	CD-13	CD-14	CD-15	CD-16
Concentration HNO_3	10%	20%	30%	40%
Time of Digestion	5 hr.	3 hr.	3 hr.	3 hr.
Thickness of Cake	1/4"	1-1/2"	3/4"	5/8"
% H_2O in Wet Cake	-	82.8%	75.9%	74.2%
Filtration Rate (filtrate and wash liquid combined) in gal./sq.ft./hr.	18.3	0.74	5.43	3.31

3x Digestions (actual HNO_3 used was 3.12 stoichiometric)

Experiment No.	CD-17	CD-18	CD-19	CD-20
Concentration HNO_3	10%	20%	30%	40%
Time of Digestion	5 hr.	3 hr.	3 hr.	3 hr.
Thickness of Cake	-	1-1/4"	3/4"	7/8"
% H_2O in Wet Cake	-	85.4%	68.1%	79.8%
Filtration Rate (filtrate and wash liquid combined) in gal./sq.ft./hr.	-	0.72	3.96	4.77

4x Digestions (actual HNO_3 used was 4.16 stoichiometric)

Experiment No.	CD-21	CD-22	CD-23	CD-24
Concentration HNO_3	10%	20%	30%	40%
Time of Digestion	5 hr.	3 hr.	3 hr.	3 hr.
Thickness of Cake	-	1-1/2"	1"	1/2"
% H_2O in Wet Cake	-	85.3%	83.7%	72.9%
Filtration Rate (filtrate and wash liquid combined) in gal./sq.ft./hr.	-	0.96	6.45	6.15

Summary and Conclusions

1. Repulp washing of the gangues from the nitric acid digestion of rich pitchblende ores is insufficient to remove the My.
2. The gangues from digestions with more concentrated nitric acid are the more readily filterable.

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