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THE VITRO MANUFACTURING COMPANY

PITTSBURGH, PENNSYLVANIA

AEC RESEARCH AND DEVELOPMENT REPORT

NYO-1154

**PROBLEMS OF REFINING URANIFEROUS
RESIDUES
TECHNOLOGY FEED MATERIALS**

Progress Report No. 20

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PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of December 1, 1952, the Research Program consisted of the following projects:

Digestion and Refining of C-Slags and Other Residues
(Project No. 1)

Investigate the applicability and refinement of various procedures for the treatment and digestion of C-Slags and other uraniferous residues for recovery of uranium values, placing emphasis on methods adaptable to the Canonsburg Plant with minimum of change of equipment.

Refining and Processing of Sodium Carbonate Residues
(Project No. 5)

Investigate methods of recovery of uranium from various residues, resulting from sodium carbonate digestion of miscellaneous uranium bearing materials, to reduce the U308 content of the residues below 0.05%.

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2. Previous Reports:

NYO-1145	March	1952 - Progress Report No. 11
NYO-1146	April	1952 - " " No. 12
NYO-1147	May	1952 - " " No. 13
NYO-1148	June	1952 - " " No. 14
NYO-1149	July	1952 - " " No. 15
NYO-1150	August	1952 - " " No. 16
NYO-1151	September	1952 - " " No. 17
NYO-1152	October	1952 - " " No. 18
NYO-1153	November	1952 - " " No. 19

PROJECT STATUS

3. Research Project - Digestion and Refining of C-Slags and Other Residues

During the program of experimentation on high grade feed material to determine whether or not the Vitro high grade feed material could be adapted to processes of a more economical nature as compared to the present established purification process, it was found that acid insolubles resulting from a hydrochloric or sulfuric acid digest of Harshaw Press Cake would be extremely difficult, if not impossible, to separate from the digest slurry by filtration. Experiments were conducted to determine whether or not calcination of the raw material at elevated temperatures prior to acid digestion would have a beneficial sintering effect, thus expediting filtration of acid insolubles.

4. Research Project - Digestion and Refining of Sodium Carbonate Residues

Experimental work concerning pitchblende carbonate residues was intensified. An extensive program was initiated to scan various processes and procedures for their applicability to pitchblende carbonate residues.

Two types of pitchblende carbonate residues are under consideration. The first type assays approximately 1.6% U3O8 and the second 0.7% U3O8. The two types vary somewhat in iron and aluminum content in addition to uranium concentration. Digestion procedures concerning mineral acids were reviewed for both types with intent to finalize digestive data and to obtain final comparative data on chemical behavior during acid digestion of both residues.

Experiments were conducted to determine whether or not the residues were amenable to organic solvent extraction techniques, based on the theory that solubilized iron may be successfully extracted from acid media by di-ethyl ether when iron is present as the chloride ion. By extraction of solubilized iron and subsequent neutralization of the aqueous phase using ammonium hydroxide it was hoped to obtain a precipitate which would be acceptable as an end product.

The possibility of a ferrocyanide selective precipitation of uranium from acid solution was considered in an additional series of experiments. The purpose of experimentation was to effect a selective precipitation of the uranyl ion using potassium ferrocyanide from an acid digest filtrate. Precipitation was to be made after reduction of solubilized iron to the ferrous state to avoid the simultaneous precipitation of contaminating "prussian blue."

Additional experiments were conducted based on the theory that ferrous iron will not precipitate as a hydroxide if complexed by the presence of sufficient quantities of ammonium chloride. In this manner, it was hoped that reduction of iron, addition of ammonium chloride, followed by neutralization of an acid digest filtrate of pitchblende residues with aqua ammonia would result in quantitative precipitation of solubilized uranium as a precipitate of high uranium content with a minimum of contamination.

Experiments were conducted surveying the possibility of extracting solubilized uranium from a mineral acid digest filtrate of the pitchblende residues as uranous phosphate by reduction of the pregnant solution with sodium hydrosulfite at low pH values. Preliminary data indicates that this may be a successful procedure in so far as the type of pitchblende carbonate residues assaying .07% U308 is concerned.

The indicated success of uranous phosphate precipitation led to the initiation of experiments designed to determine optimum conditions of precipitation necessary for quantitative extraction. A preliminary investigation of sodium hydrosulfite requirements, the most desirable pH of precipitation, and the benefits of the addition of excess PO_4 ion was completed.

B. DIGESTION AND REFINING OF C-OXIDE AND OTHER RESIDUES

Research Project No: 1

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

5. Filtration of Acid Insolubles Resulting From A Hydrochloric or Sulfuric Digest of Harshaw Press Cake

During the process of experimenting on Vitro high grade feed materials to determine the feasibility of shorter and more economical means of refining these materials, it was noted that acid insolubles resulting from a hydrochloric or sulfuric acid digest of Harshaw Press Cake exhibited slime characteristics and as a consequence were almost impossible to filter. The normal means of preparing Harshaw Press Cake for production purification consists of a process of drying and calcination at temperatures approximating 1100° F, in order to remove volatile organic contaminants such as tributyl phosphate. The fired material, after comminution is processed in the usual manner. Acid insolubles resulting from a production digest of Press Cake are not normally removed from the digest slurry until after neutralization of the digest with sodium carbonate. This procedure carried out in the established Vitro process is satisfactory in effecting maximum filtration of insolubles since they apparently exhibit different characteristics in the presence of alkali. However, if the proposed treatment consisting of hydrochloric acid digestion and precipitation of ammonium

di-uranate from a clarified filtrate followed by calcination of the di-uranate precipitate to a black oxide at 800°C is to be effective as regards press cake, it is necessary to determine some means of expediting removal of acid insolubles from the acid digest slurry. Samples of Harshaw Press Cake were taken from production lots and calcined at varying temperatures up to 1600°F to determine if elevated temperatures would have a sintering effect on the raw material which would eliminate the formation of slimes or gel during acid digestion. Completed experimental data indicates that if the material is calcined at 1400°F for one hour prior to acid digestion, the acid insolubles resulting from hydrochloric acid digestion may be easily and readily removed by normal means of filtration without the assistance of artificial filtering aids. Filtration of the insoluble residue was rapid and easily washed uranium free. Acid insolubles resulting from acid digestion of the sintered material contained less than .05% U_3O_8 . Complete experimental data may be found in Table 1.

In general, with reference to experimentation on optimum procedures for the refining of high grade feed materials, sufficient data has been accumulated on the laboratory level to carry such procedures into the pilot or semi-works phase. Unless unknown circumstances should indicate additional laboratory development on the materials under consideration, or similar materials, no further experimentation is presently planned.

C. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project No: 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

5. Mineral Acid Digestion of Sodium Carbonate Residues Resulting as a By-Product of Pitchblende Purification

Pitchblende carbonate residues may be categorically divided into two groups for experimentation. These two groups represent the entire stockpile of this type of sodium carbonate residues at the Vitro installation. The first group of residues contain approximately 1.60% U3O8, plus aluminum as the principal contaminant with a proportionately smaller amount of iron and silica and vanadium. This group of residues is identified as Pitchblende Carbonate Residue "A". The second group of residues has a uranium content of 0.69% U3O8 and contains iron as the major contaminant with proportionately smaller amounts of aluminum, silica, vanadium, etc. The second group of residues is identified as Pitchblende Carbonate Residue "B". Since the two groups of residues contain proportionately different amounts of uranium and variable quantities of aluminum and iron they are considered separately and experimental data is recorded in this manner.

10-67-X

Pitchblende Residue A.

Additional experiments concerning mineral acid digestion of this residue were completed. In general, all data obtained tends to confirm previous experimentation of the same nature. 100 gram samples of the dried residue were pulverized to less than 100 mesh and digested in sulfuric acid at pH values ranging from .0 to 0.5 in the presence of 1% (w/v) sodium nitrate. Time of digestion was from 1 - 4 hours at a temperature range of 80° C - 90° C. Acid insolubles resulting from the procedure were voluminous, of a slimy nature, and very difficult to filter. The uranium level of sulfuric acid digest insolubles remained well in excess of maximum specifications for discard residues, containing more than .30% U308. This data tends to confirm previous data and we may assume that a sulfuric acid treatment is undesirable and inapplicable to this residue.

Digestion of this residue in a hydrochloric acid media was somewhat more successful. 100 gram samples of the dried residue were pulverized to less than 100 mesh, slurried in water at a ratio of 4:1 and digested in commercial hydrochloric acid at pH values ranging from 0.0 to 0.5 in the presence of 0.5% (w/v) sodium chlorate for periods of time extending to four hours at temperatures ranging between 80° C - 90° C. This procedure, as indicated from previous data, will be satisfactory in solubilizing contained uranium and the digest slurry will lend itself to removal of acid insolubles by filtration. The uranium content of the insoluble residue resulting from the HCl digest treatment contained less than .05% U308, and may be considered satisfactory for a discard residue. Insolubles were found to represent 6 to 8% of the original volume. Representative data may be found in Table II-A.

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Pitchblende Residue B

Additional experiments were conducted concerning mineral acid digestion of this residue. Sulfuric acid was again found to be an unsatisfactory digest media. 100 gram samples of the raw material were dried, pulverized to less than 100 mesh, slurried in water at a ratio of 4:1 and digested in sulfuric acid at pH values ranging from 0.0 to 0.5 for periods of time up to four hours at temperatures ranging between 80 and 90° C. The insoluble residue resulting from this treatment was easily removed by filtration, but was found to contain uranium considerably in excess of discard specifications. The residue approximated 75% of the original volume and had an average assay of .26% U308. However, hydrochloric acid digestive techniques, as in the case of Pitchblende Residue A, were found to be entirely satisfactory as regards successful extraction of the contained uranium. 100 gram samples of the dried material were pulverized to less than 100 mesh, slurried in water at a ratio of 4:1 and digested in commercial hydrochloric acid at pH 0.0 - 0.5 in the presence of 0.5% (w/v) sodium chlorate at 80° C for periods of time up to four hours. Acid insolubles averaged approximately 10% of the original volume and contained less than .05% U308. Calcination of the raw material prior to digestion did not markedly alter the chemical characteristics of the residue during mineral acid digestion. Representative data may be found in Table II-B.

6. Refining of Pitchblende Residues by a Process of Hydrochloric Acid Digestion and Di-Ethyl Ether Extraction of Iron

Experiments were concluded on pitchblende residues based on the theory that iron may be extracted as a soluble chloride from acid solution by the use of an non-miscible organic solvent

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such as di-ethyl ether. Uranium is not extracted as the chloride ion. It was theorized, that if this were feasible the extraction of ferric chloride from a hydrochloric acid digest filtrate of pitchblende carbonate residues using ether, followed by a precipitation of uranium from aqueous phase might result in a precipitate which would be an acceptable product. It was demonstrated by preliminary experimentation that such a procedure would be impractical. It was immediately evident that iron was present in such large quantities that ether would have to be used in such volume as to make the procedure economically impractical. In addition, the presence of large quantities of sulfate and phosphate ion did not permit adequate extraction of iron even when large quantities of ether were consumed. A further difficulty that developed throughout the process was the tendency of the ether phase to gel due to the presence of large quantities of siliceous matter which passed into the organic phase. No further experimentation of this nature is planned.

7. Precipitation of Uranium From a Hydrochloric Acid Digest Filtrate of Pitchblende Residues Using Potassium Ferrocyanide

Scoping experiments were completed designed to determine if uranium could be selectively and quantitatively precipitated from a hydrochloric acid digest filtrate of pitchblende carbonate residues by the addition of potassium ferrocyanide. The reaction should procede in the following manner:

The resultant precipitate could then be processed to sodium di-uranate by the use of sodium hydroxide as follows:

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In order to avoid a simultaneous precipitation of the ferric salt of ferrocyanic acid it was necessary to prepare the solution for precipitation by reducing iron in solution to the ferrous state. It was hypothesized that iron could be reduced in the presence of uranium, without reduction of uranium, by the addition of sodium thiosulfate catalyzed by the addition of a small quantity of copper. Experiments conducted in this manner on hydrochloric acid digest filtrates of pitchblende carbonate residues were only partially successful. Although it was possible to remove uranium using this technique it was not possible to obtain quantitative reduction of solubilized iron. This was probably due to exposure to air as the solution was agitated. As a result, uranium precipitation was accompanied by the precipitation of "prussian blue". Contamination of the resultant precipitate was such that the procedure was ineffectual. Uranium content of the residue did not exceed 4 - 5% U308. However, further investigation is indicated and additional experiments may be conducted to determine if this technique may be developed into a successful means of separation.

8. Precipitation of Uranium From A Hydrochloric Acid Solution of Pitchblende Residues Using Aqua Ammonia After Complexing Reduced Iron With Ammonium Chloride

Experiments were completed based on the theory that iron, when reduced to the ferrous state and complexed with sufficient ammonium chloride, will not precipitate in the presence of ammonium hydroxide. Normally, ammonia produces in neutral solutions an incomplete precipitation of ferrous hydroxide. The reaction moves to the left when excess ammonium chloride is present, as follows:

It was assumed that should this procedure prove feasible as regards a hydrochloric acid digest filtrate of pitchblende residues, uranium could be precipitated as ammonium di-uranate without the usual contamination from precipitated iron. The resultant residue would then be easily purified to an acceptable end product. Filtrates from hydrochloric acid digestive procedures were used for experimentation. Solubilized iron was reduced to the ferrous state by the addition of sodium thiosulfate. Ammonium chloride was added in excess of theoretical requirements. The pregnant solution was neutralized to pH 6.0 with ammonium hydroxide. Only partial success was indicated. As illustrated by other experimentation, reduction of solubilized iron to the ferrous state was far from complete, probably due to exposure to air during agitation. The residue resulting from neutralization with aqua ammonia was heavily contaminated by both iron and aluminum. Uranium content of the precipitate did not exceed 3.5% U308. The economics of this procedure and the poor results of preliminary experimentation do not indicate that additional experiments will be practical.

9. The Precipitation of Uranous Phosphate From the Hydrochloric Acid Digest Filtrate of Pitchblende Carbonate Residues

Preliminary experiments were initiated to estimate the feasibility of adapting pitchblende carbonate residues to a process of uranous phosphate precipitation from a clarified hydrochloric acid digest filtrate by reduction of the uranyl ion with sodium hydrosulfite. The P_2O_5 content of the residues is sufficiently high to effect complete precipitation of uranium without further addition of phosphates. Preliminary experiments indicate a possibility of a practical application. Since this type of a precipitation is influenced by soluble aluminum content of the pregnant liquor and aluminum content of Pitchblende Carbonate "A" and "B" is not the same, results for the two different types of pitchblende residues are considered separately.

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Pitchblende Carbonate Residue 'A'

100 gram samples of this residue assaying 1.6% U308 were dried, pulverized to less than 100 mesh, slurried in water at a ratio of 4:1 and digested in commercial hydrochloric acid at pH 0.5 in the presence of 0.5% (w/v) sodium chlorate for one hour at 80° C. Acid insolubles were removed by filtration and the clarified filtrate used for experimentation. Sodium hydrosulfite was added to the pregnant liquor in excess of theoretical requirements at pH 2.0. The precipitate that formed was both voluminous and difficult to filter, indicating that a disproportionate amount of solubilized aluminum with resultant complexing activity. Analysis of the clarified filtrate indicated a discard effluent assaying from 0.025 - .10 U308. Recovery, of course, was unsatisfactory ranging from 60 - 90% total yield. The uranous phosphate residue was heavily contaminated with aluminum and averaged only about 3 - 4% U308. It is not immediately apparent that pitchblende residue "A" will be amenable to this treatment. However, additional experiments are indicated and a survey of the possibility of successful reduction using metallic iron or aluminum is planned. Preliminary data may be found in Table III-A.

Pitchblende Carbonate Residue "B"

Preliminary experiments were completed on the precipitation of uranous phosphate from a pregnant hydrochloric acid digest filtrate of pitchblende residue "B". Initial experiments indicated successful and satisfactory extraction of solubilized uranium. 100 gram samples of residue "B" were slurried in water at a ratio of 4:1, digested in HCl at pH 0.5 for one hour at 80° C, oxidized with 1% (w/v) NaClO₃ and filtered. The clarified filtrate was reduced with Na₂S₂O₄ · 2 H₂O at pH 2.0 and the resultant precipitate separated by filtration. Discard effluents averaged .005% U308 indicating a total yield of 95%.

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Due to the satisfactory nature of preliminary data a series of experiments was conducted to obtain preliminary data on optimum pH of precipitation, sodium hydrosulfite requirements and phosphate ion requirements. 100 gram samples of Residue "B" were slurried in water at a ratio of 4:1, digested in HCl at pH 0.5 for one hour at 80° C, oxidized with 1% (w/v) NaClO₃ and filtered. The insoluble residues were acceptable for discard. Filtrates were used for experimentation. Uranous phosphate was precipitated from the pregnant solution over a pH range of 0.5 - 2.0 using quantities of Na₂S₂O₄ · 2 H₂O ranging from 2.0 gm. - 8.0 gm. In some instances, additional phosphate was added in the form of Na₃PO₄ prior to precipitation. In general, results are encouraging although nothing conclusive may be stated at this time. An extensive program of experimentation is planned to correlate all factors leading to a satisfactory procedure. Preliminary data is presented in Table III-B.

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EXHIBITS

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TABLE I

HYDROCHLORIC ACID DIGESTION OF H RSHAW PRESS CAKE

MLSH: 85% -100

	<u>EXPERIMENT NUMBER</u>					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Temperature of Calcination (°C)	1100	1200	1300	1400	1500	1600
Weight of Sample (gm.)	500	500	500	500	500	500
Weight of H ₂ O (gm.)	2000	2000	2000	2000	2000	2000
Weight of HCl (gm.)	425	425	425	425	425	425
Digest Time (hr.)	3	3	3	3	3	3
Digest Temperature (°C)	80	80	80	80	80	80
pH of Digestion	0.0	0.0	0.0	0.0	0.0	0.0
Filtration Time (min.)	Non- Filterable	Non- Filterable	48.0	8.5	8.0	8.5
Acid Insolubles (gm.)	--	--	214	208	205	207
Acid Insolubles (% U308)	--	--	.15	.050	.055	.035

TABLE II-A

MINERAL ACID DIGESTION OF PITCHBLEND CARBON- TF RESIDUE A'

ASSAY: 1.6% U308

	<u>EXPERIMENT NUMBER</u>							
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
Weight of Sample (gm.)	100	100	100	100	100	100	100	100
Weight of H ₂ O (gm.)	400	400	400	400	400	400	400	400
Weight of H ₂ SO ₄ (gm.)	--	--	--	--	165	155	165	165
Weight of HCl (gm.)	280	270	275	275	--	--	--	--
Time of Digest (hr.)	1	2	3	4	1	2	3	4
pH of Digest	0.0	0.5	0.20	0.15	0.0	0.5	0.20	0.14
Temperature of Digestion (°C)	80	80	80	80	80	80	80	80
Acid Insolubles (gm.)	6.0	8.0	6.5	7.2	130	132	130	135
Acid Insolubles (U308)	.050	.032	.032	.055	.31	.33	.33	.33

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TABLE II-B
MINERAL ACID DIGESTION OF PITCHBLEND CARBONATE RESIDUE 'B'

ASSAY: 0.69% U308

	<u>EXPERIMENT NUMBER</u>							
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
Weight of Sample (gm.)	100	100	100	100	100	100	100	100
Weight of H2O (gm.)	400	400	400	400	400	400	400	400
Weight of HCl (gm.)	235	235	220	230	--	--	--	--
Weight of H2SO4 (gm.)	--	--	--	--	145	132	135	135
Time of Digest (hr.)	1	2	3	4	1	2	3	4
pH of Digest	0.0	0.0	0.5	0.35	0.0	0.55	0.35	0.30
Temperature of Digest (°C)	80	80	80	80	80	80	80	80
Acid Insolubles (gm.)	6.5	8.5	10.0	9.2	75.5	75.0	74.6	76.2
Acid Insolubles (U308)	.032	.055	traces	.055	.26	.31	.22	.28

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TABLE III -

PRECIPITATION OF URANOUS PHOSPHATE FROM AN HCl DIGEST FILTRATE OF PITCHBLEND
RESIDUE

ASSAY: 1.6% U308

	<u>EXPERIMENT NUMBER</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of Sample (gm.)	100	100	100	100
Weight of H ₂ O (gm.)	400	400	400	400
Weight of HCl (gm.)	270	270	270	270
pH of Digestion	0.5	0.5	0.5	0.5
Time of Digest (hr.)	1	1	1	1
Temperature of Digestion (°C)	80	80	80	80
pH of Precipitation	2.0	2.0	2.0	2.0
Na ₂ S ₂ O ₄ · 2 H ₂ O added (gm.)	2.0	3.0	4.0	5.0
Residue (gm.)	33.0	31.5	31.0	35.0
Residue (% U308)	3.0	3.86	3.64	3.75
Discard Effluent (% U308)	0.10	.049	.046	.024
Recovery (%)	60.0	80.4	84.6	90.0

TABLE III-B

PRECIPITATION OF URANOUS PHOSPHATE FROM AN HCl DIGEST FILTRATE OF PITCHBLEND

RESIDUE "B"

ASSAY: 0.69% U308

	<u>EXPERIMENT NUMBER</u>									
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>
Weight of Sample (gm.)	100	100	100	100	100	100	100	100	100	100
Weight of H ₂ O (gm.)	400	400	400	400	400	400	400	400	400	400
Weight of HCl (gm.)	220	220	220	220	220	220	220	220	220	220
pH of Digestion	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Time of Digest (hr.)	1	1	1	1	1	1	1	1	1	1
Temperature of Digestion (°C)	80	80	80	80	80	80	80	80	80	80
pH of Precipitation	0.5	1.0	1.5	2.0	0.5	1.0	1.5	2.0	2.0	2.0
Na ₂ S ₂ O ₄ · 2 H ₂ O Added (gm.)	2.0	2.0	2.0	2.0	5.0	5.0	5.0	8.0	2.0	4.0
Na ₃ PO ₄ Added (gm.)	--	--	--	--	--	--	--	--	2.0	2.0
Residue (gm.)	10.0	10.0	11.0	10.5	10.5	10.5	11.0	11.0	10.0	11.0
Residue (% U308)	3.30	4.24	6.22	6.40	3.75	6.25	6.10	6.30	6.45	6.15
Discard Effluent (% U308)	.061	.046	.006	.001	.049	.01	.0001	.001	.007	.005
Recovery (%)	39.0	54.0	94.0	99.0	51.0	90.0	99.9	99.0	93.0	95.0

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