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Influence of time and temperature in the roasting of sulphides

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THESIS

FOR

The Degree Bachelor of Science
in
Chemistry and Metallurgy.

~~~~ Roger H. Hatchett. ~~~~
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INFLUENCE OF TIME AND TEMPERATURE
IN THE
ROASTING OF SULPHIDES.

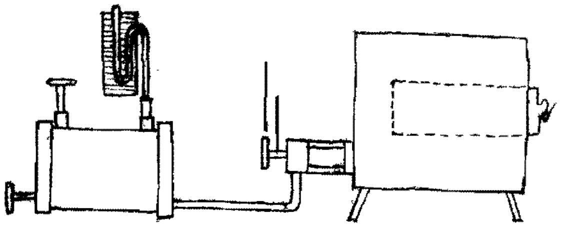
The following experiments were made by Mr.E.C.Bierbaum and myself:

The original object of this work was to determine the relation between the quantities of oxide and sulphate formed in roasting a sulphide under known conditions of temperature, time and supply of air. But difficulties arose in constructing apparatus, which in the short time allotted could not be overcome. These are mentioned in description of apparatus, and were of such a nature that the relations between products and conditions are for the most part qualitative.

Apparatus for Roasting.

The first apparatus tried for roasting the ores, was a square sheet-iron tube, having the ends fitted with practically air tight doors with a tube attached for admitting and removing air. This was fastened lengthwise in a rectangular sheet iron box which served to prevent sudden cooling of the tube. The box was placed over three Bunsen burners so that the tube was directly over the flame, and the ore to be roasted was placed inside this tube. Air was to be aspirated through this apartment and drawn out through a solution of sodium hydrate to measure the sulphur dioxide given off, as a check on the analysis of roasted products. Although the temperature of the Bunsen flame was between 500 and 600 degrees it was impossible to get more than 350 degrees C. inside the tube. Galena was heated at this temperature with plenty of air for three or four hours, without being appreciably changed. Failure to get sufficient high temperature with Bunsen burners prevented the use of what seemed the best arrangement of apparatus, and at the same time the attempt to measure the supply of air.

The apparatus then decided upon was a small Hoskins gasoline muffle furnace as shown in accompanying sketch.



To regulate the temperature the tank was fitted with a pressure gauge and a pointer indicated to what extent the valve was opened. This arrangement was used for all the roasting done.

The muffle was left open during the roasting so that there was always free access of air, though with different temperatures the quantity varied.

The ores used were galena and blende as nearly pure as could be obtained. These were pulverized to pass through a 60 mesh sieve, and each portion roasted in 2 1/2 inch fire clay scorifiers, noting in each case the temperature and time of roasting.

After three or four hours use the burner of the furnace began to fill with a carbonaceous deposit from the decomposition of the gasoline and with the gauges set to read the same, the temperature was different.

This difficulty prevented the accurate determination of temperature of each roast, as it was intended to do this after roasting, by removing any lead fumes and repeating the conditions. Lead fumes had to be removed because temperatures were measured with a pyrometer.

Method of Analysis of Roasted Galena.

The problem presented for analysis of the roasted galena was the complete separation of lead oxide, lead silphate, and lead sulphide. No account of a method for this separation could be found, so some qualitative experiments were made with solvents, for the three compounds.

These experiments indicated that a good method if not the best one for separating oxide, sulphate and sulphide of lead is the following:

First. Dissolve lead oxide in a 10-15 % solution of acetic acid- either cold or hot.

Second. Dissolve lead sulphate in hot concentrated ammonium acetate.

Third. Dissolve the remaining sulphide as in a regular analysis.

The advantages this scheme offers are:

First. It is rapid, allowing all but the total sulphur determinations to be made volumetrically.

Second. The solution of Oxide and sulphate is ready for titration with a standard solution of molybdate, and the nitric acid solution of the sulphide is easily prepared for titration, by precipitating the lead as sulphate and dissolving in amonium acetate.

Third. The separation is apparently complete.

The accuracy of the method was tested by making mixtures of galena, lead sulphate and oxide. The galena was the same as that used for roasting and contained 83.3% lead. The oxide contained 91.6 % lead and the sulphate was pure.

A second series of tests were made with the mixture unknown to the analyst at the time the analyses were made. Mixtures prepared by Mr. Bierbaum were analysed by myself and vice versa.

The following tables show the results of these analyses. The first table was obtained from known mixtures of 100 and 200 milligrams of each compound. The second and third tables are from unknown mixtures. Weights are in milligrams.

	PbO		$PbSO_4$		PbS.	
Trial	Cal.	Found	Cal.	Found	Cal.	Found.
1	98.7	98.6	100	98.6	96.5	
2	98.7	99.3	100	99.4	96.5	
3	197.4	197.4	200	194.	193	194.6
4	197.4	197.4	200	194.6	193.	192.

Mixtures Analyzed by Mr. Bierbaum.

	PbO		$PbSO_4$		PbS	
Mixture	**Cal.**	Found	Cal.	Found	Cal.	Found.
1	98.7	100.6	300	295.4	96.5	96.5
2	197.4	196.1	170	168.9	144.7	143.7
3	345.4	344.2	120	118.8	193.0	193.0

Mixtures analyzed by myself.

1	345.4	344.9	225.	224.	103.6	101.9
2	109.5	110.5	300	298.	220.1	220.4
3	196.4	198.3	130	129.9	328.6	329.6

These mixtures were made to contain the compounds to be separated, in varying proportions and since the majority of the results are within the limit of experimental error, it may be concluded that the method outlined is accurate, and a good one for analysis of roasted galena.

Roasted Galena.

Fifteen grams of Galena were used in each roast. This made a layer in the dish 1/2 inch to 3/4 inch thick, which allowed almost the entire amount to be exposed to the air.

No stirring was attempted. Two samples were roasted together, the first of each set being in the front part of furnace.

The following is a table giving the time, temperature and analysis of several samples of roasted galena:

			Products.			Total	Total S	Ratio	Ratio	
Sample	Temperat.	Time.	PbO	$PbSO_4$	PbS	S	as Sulphide	$PbO:PbSO_4$	PbO:PbS	
1	Fused	3 hrs	274.	87.	49.5	15.8	6.6	3:1	55:1	Met.Pb
2	Semifused	3 "	254.	96.	51.0	12.2	6.8	25:1	5:1	Met.Pb
3	Not fused	3 "	164	251.	77.0	36.9	10.4	.66:1	2:1	
4	750 ° C	2 "	151	79	24.5	41.0	32.6	2:1	.6:1	
5	700° C	2 "	187	99	200.	37.0	26.6	2:1	.9:1	
6	750° C	3 "	185	273	32.	33.0	4.2	.66:1	6:1	Met.Pb
7	700° C	3 "	204	246	38.	31.0	5.7	.75:1	6:1	Met.Pb
8	750° ?	4 "	156	213	124	39.0	16.6	.75:1	1;2:1	
9	700° ?	4 "	155	270	68	37.0	9.1	.55:1	2;1:	
10	750 850	4 "	279	122	85.	24.0	11.3	2:1	3;3:1	
11	700 800	1 " 4 "	187	291	00	30.7	00.0	.66:1		
12	750 850	1 " 4 "	308	132	00	13.9	00.0	2.3:1		
13	750 850	1 " 4 "	215	257	00	27.0	00.0	.8:1		
14	750 850	1 " 4 " 1	252	217	00	22.0	00.0	1.1:1		

One, two and three were roasted together, 1 in back, 2 in center and 3 in front part of muffle. This shows at the outset what a difference ~~the~~ there is in the products at different temperatures. Temperature was not measured for these but for the rest, Three determinations gave practically the same result.# The reason for this limit is that below 700 degrees the roasting is very slow, and above 850 degrees the products are all fused. #The table shows only about 150 degrees variation in temperature

The difference of temperature between the front and back of the muffle was near 50 degrees C. so this has been recorded as the difference in each case. The reason 8 and 9 show lower oxide for 4 hours roast than 6 and 7 do for 3 hours, at what is given at the same temperature is, that for 6 and 7 the burner had just been cleaned while for 8 and 9 it was somewhat choked, so with gauges set the same, different temperatures resulted.

Nos. 10-12 and 14 were roasted in the same position in the muffle at different times, with gauges set the same. Similarly Nos. 11 and 13 should agree in composition being in same position in muffle but not the same as 10,12 and 14. These last named five were roasted for four hours at about 750 degrees C. when considerable sulphate was formed as in 8 and 9. Then the temperature was raised just to fusion point (800 -850 degrees). This prevents metallic lead from forming as it did in Nos. 1,2,6 and 7 i.e., the reaction between lead oxide and lead sulphate does not take place at so low a temperature as does the reaction between lead oxide and lead sulphide. This accounts for slow roasting at the beginning in practice.

Another indication of this is, that in Nos. 1,2,6 and 7 metallic lead was formed and sulphide is very low. By the method employed for 10-12 and 14 a complete change is effected in the sulphide.

To verify these conclusions a determination was made of total Sulphur in the residue from treating with ammonium acetate, in Nos. 6,7,8 and 9. and also a total Sulphur determination was made of Nos. 11,12,13 and 14, which according to our scheme of analysis showed no Sulphide.

The following tables show the results of these tests:

	PbS	
Sample	S as found in our scheme of analysis	Total S determ. in Residue from $NH_4C_2^{H3}$ Solution
6	4.2 milligrams	4.1 milligrams
7	5.0 "	4.98 "
8	16.6 "	16.6 "
9	9.2 "	9.06 "

Sample	S as PbS found in our scheme of analysis	Total S determ. in Residue from $MH_4C_2H_3$ Solution.
11	30.78 milligrams	29.1 milligrams
12	13.92 "	12.8 "
13	27.00 "	27.5 "
14	22.90 "	24.0 grams.

This includes all analyses made.

Some of these samples contained lead silicate and since the total sulphur determination in the residue from treating with ammonium acetate agree with Sulphur found as lead sulphide in the regular analyses, the lead silicate must be decomposed by the acetic acid. Similarly the total sulphurs agreeing with Sulphur as sulphate found in regular analysis prove the absence of lead sulphide.

The influence of iron, zinc, etc. was not investigated.

Roasting Blende.

The same method of roasting at different temperatures was tried with nearly pure sphalerite. Samples were taken at various stages of the roast and at different temperatures. It was found that after about 2 1/2 to 3 hours roasting at 750 degrees-800 degrees C the Sulphide was completely changed to oxide and in no case could zinc sulphate be found. This was tried by boiling with water and digesting with sodium carbonate to decompose any basic zinc sulphate that might be insoluble if formed.

This work agrees with practice at Joplin, Mo. where it is said no sulphate is formed in roasting. But statements are made in text books# that zinc sulphate is formed from zinc sulphide by slow roasting.

Crookes and Rohrig Metallurgy of Lead, Silver, Zinc and. p412

Graham-Otto-Lehrbuch der Chemie. Vol LLL p 809

Though this work has brought out no striking relations between products and conditions it indicates that such exist, and that they might have been established for Galena if sufficient time had remained to construct apparatus so that the temperature could be regulated at will, and kept constant.

It has further brought out an accurate method for the analysis of roasted galena, and the different behavior of galena and sphalerite on roasting.