

GAS ANALYSES.

Experiment number.	Sample number.	Oxygen. Per cent.	Ethylene. Per cent.	Carbon monoxide. Per cent.	Methane. Per cent.	Hydrogen. Per cent.
20	1	15.6	9.8	1.3	—	—
21	1	18.0	16.0	4.4	—	—
21	2	79.6	—	—	—	—
22	1	66.7	—	1.0	—	—
25	1	17.2	1.0	10.0	—	—
32	1	3.06	1.96	6.55	44.11	17.90
32	2	3.18	2.72	12.84	24.40	35.88
33	1	77.5	—	3.2	—	—
34	1	18.5	—	1.0	—	—
34	2	2.4	3.1	9.5	31.2	28.9
34	3	41.0	1.0	4.0	—	—
36	1	28.2	3.6	—	—	—
36	2	75.4	5.0	9.6	5.00	0.8
36	3	39.4 ¹	14.2	—	—	—
36	4	83.5	7.2	4.3	—	—
37	1	31.0	3.6	3.6	—	—
37	2	60.8	5.8	—	—	—
37	3	66.7	4.6	2.8	—	—
37	4	77.4	2.0	2.6	—	—

NOTES ON THE DETERMINATION OF SULPHUR IN PIG IRON.

BY M. J. MOORE

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MUCH has been said regarding the inaccuracy of the volumetric method for the determination of sulphur in pig iron, and many good reasons too well-known to mention here, have been given by way of explanation. I will refer, however, to one article by Phillips² which illumines some of the dark places admirably.

The writer is not aware that any mention has ever been made that physical treatment was responsible for a great portion of the error, such treatment of course, changing the chemical composition of the iron. It is the practice of nearly all if not all, steel and furnace plants to pour the molten metal into water, this being called a "shot sample." The shot thus formed are taken to the laboratory and crushed in a steel mortar and passed

¹ It was found that the pyrogallic acid was exhausted; hence this discrepancy.

² This Journal, 17, 891.

through a sixty or eighty mesh sieve, the powder being subsequently analyzed for silicon and sulphur.

I was brought face to face with the sulphur problem some little time ago and in order that the reader may appreciate my plight some explanation will be necessary.

We were called upon to take samples every hour from the cupolas and mixer, analysis being made for silicon and sulphur on each test continuously for the twelve hours. Our practice at this time was to catch a small test in a sand mold, but it was found necessary to change the mode of sampling because the cupola test was nearly always too hard to drill owing to low silicon and high sulphur which rendered the metal white. The mixer tests, however, could be drilled without any trouble. In order to get around this difficulty we caught shot samples from the cupolas but still continued sampling from the mixer in the old way. Continuing this for twenty-four hours and comparing results it was found that the mixer samples carried more sulphur, apparently, than the cupola samples when the reverse should have been the case especially when low sulphur furnace metal was charged into the mixer. So in order to get comparative results shot samples were caught at the mixer. The next problem that presented itself was to find why there was such an apparent increase of sulphur from the mixer metal to the steel.

To ascertain if there was any difference in sulphur when simply the volumetric method was used, between a drilled sample and a shot sample, samples were caught from the mixer ladle while the metal was being poured into the converter. A portion of the metal was poured into water and the balance into a sand mold. The shot samples were reduced in the usual manner and passed through an eighty mesh sieve and the sand samples were drilled. Determinations for sulphur were made on each, both by the volumetric and gravimetric methods, the latter method according to Blair's "Analysis of Iron and Steel." The results are given herewith :

JOLIET WORKS, ILLINOIS STEEL COMPANY.

Analysis of mixer metal for sulphur, showing that the volumetric method does not give all the sulphur, and that the error is greater in shot samples than in drilled samples.

Shot Samples from Mixer.

Date.	Blow No.	Volumetric method.	Gravimetric method.	Difference.	Sulphur in residue.	Volumetric sulphur plus residual.	Column 2 minus column 5.	Sulphur in steel.	Remarks.
1899.		1	2	3	4	5	6		
Jan. 9 ..	1026	0.056	0.081	0.025	0.005	0.061	0.020	0.083	Samples were caught from same test ladle.
" 16 ..	2123	0.064	0.096	0.032	0.008	0.072	0.024	0.103	
" 16 ..	2124	0.067	0.095	0.028	0.007	0.074	0.021	0.104	
" 16 ..	2125	0.070	0.103	0.033	0.008	0.078	0.025	0.106	
" 16 ..	2126	0.073	0.103	0.030	0.006	0.079	0.024	0.106	
Mean ..		0.066	0.096	0.030	0.007	0.073	0.023	0.100	

Sand Samples from Mixer.

Date.		1	2	3	4	5	6		
1899.									
Jan. 9 ..	1026	0.068	0.081	0.013	0.0113	0.079	0.002	0.083	Note difference between the volumetric method in column 1.
" 16 ..	2123	0.089	0.098	0.009	0.007	0.096	0.002	0.103	
" 16 ..	2124	0.087	0.101	0.014	0.009	0.096	0.005	0.104	
" 16 ..	2125	0.087	0.103	0.016	0.008	0.095	0.008	0.106	
" 16 ..	2126	0.088	0.103	0.015	0.005	0.093	0.010	0.106	
Mean ..		0.084	0.097	0.013	0.008	0.092	0.005	0.100	

Referring to the mean results we find in column 1 the difference by the volumetric method to be 0.018. This is enough to condemn the practice of taking shot samples.

In column 2 the total sulphur by the gravimetric method is practically the same in each case. In column 3 one exceeds the other by 0.017. In column 4 the residual sulphur in each case is practically the same and in column 6 we see that all the sulphur under shot samples has been accounted for except 0.023 per cent., while under sand samples we are obliged to account for but 0.005 per cent. Or, in one case there is an actual loss of 23.95 per cent., while in the other but a trifle over five per cent.

The results herein given I consider conservative as both

Messrs. Blair and Dudley have pointed out that all the sulphur is not oxidized by the aqua regia method. The writer feels that too much stress cannot be brought to bear on the sulphur question in pig iron as no concordant results can be brought about from the pig iron to the steel.

One of the prominent eastern mills, to account for the error, uses a factor which is far ahead of reporting 100 per cent. of the results wrong as is the case with the evolution method. In conclusion, the writer earnestly invites any criticism if he has made misstatements. His motive has only been to help along the science.

Note.—Since writing the above more experimenting along this line with iron low in sulphur, say No. 1 Bessemer, has brought out the fact that the error between the gravimetric and volumetric methods is not so apparent until about a No. 2 grade is reached.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE PENNSYLVANIA STATE COLLEGE AGRICULTURAL EXPERIMENT STATION.]

A CONTRIBUTION TO THE CHEMISTRY OF BUTTER-FAT.

BY C. A. BROWNE, JR.

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III. THE CHEMISTRY OF RANCIDITY IN BUTTER-FAT.

THE phenomena attending the development of rancidity in fats have attracted the attention of chemists for many years; even before the beginning of the present century, ere Chevreul had begun his classical "recherches chimiques sur les corps gras," and when the chemical constitution of fats was still unknown, it had been surmised that the physical changes which oils and fats underwent on long keeping were simply the results of oxidation; and this is the view most generally accepted at the present time, notwithstanding the fact that many eminent chemists in the past have sought to explain the development of rancidity in other ways.

From the very complexity of its composition we would suppose butter-fat to be a rather unstable body; if butter-fat is sealed up and preserved in a cold, dark place, it will retain its normal character and appearance for many months, but if kept in a