(10-A) Item #18 Tom 132 753

Analytical Results and Methods for Gas Purification Catalysts

The gas purification catalyst is either natural iron ore or the so called Lauta-mass, a residue from the manufacturing of aluminum from Bauxite.

Translator

Analysis of the spent mass from tower #3 withdrawn: January 26, 1938. Sulfur content according to the balance: 112 too.

Tray	Date		$\%H_2O$	%S Dry Mass	%S Moist mass	%S/SO$_4$	$\%FeSO_4$
1	Jan.	27	7.0	53.9	50.1	1.24	
2	"	27	6.5	54.0	50.5	1.29	
3	"	29	6.5	55.5	51.9	1.31	
4	"	31	7.0	51.8	48.2	1.19	
5	"	31	5.5	56.0	52.9	1.30	
6	Feb.	1	6.5	53.3	49.8	1.16	
7	"	1	6.5	54.0	50.5	1.17	
8	"	2	7.0	53.2	49.9	1.15	
9	"	2	8.0	54.3	50.0	1.25	
Average			6.5	54.3	50.7	1.21	

The mass from tray #2 has ignited during the shipping,
The mass from tray #3 had to be quenched after arrival at the chemical factory: von Heydn.

Analyses of spent iron ore
Tower 2

Tray	H_2O %	Sulfur content % moist	% dry	Loaded 1940	shipped 1940	Freight Car#
1	4.5	53.61/53.40	56.03/55.92	21.5	21.5	Nurnberg - 4933
1+2	4.2	57.71/57.51	60.24/60.03	"	"	Schwerin - 3216
2	5.0	53.03/53.09	55.82/55.86	"	22.5	Deutsch - 388778
3	5.5	51.29/51.45	54.27/54.44	22.5	"	Schwerin - 10909
4	5.0	51.92/52.00	54.65/54.74	"	"	Schwerin - 4316
4+5	6.5	50.78/50.76	54.31/54.29	"	24.5	Deutsch - 342861
5	5.75	53.34/53.34	57.65/57.65	"	"	Schwerin - 4952
6	5.0	56.50/56.51	59.47/59.48	23.5	"	Schwerin - 17641
7	6.0	51.04/51.24	54.28/54.51	"	"	Nurnberg - 26408
7+8	6.0	48.71/48.80	51.82/51.91	24.5	25.5	Schwerin - 1929
8	4.25	52.17/52.01	54.49/54.33	"	"	Deutsch - 381831
9	11.5	46.13/45.96	52.12/51.93	25.5	"	Deutsch - 321299
10	5.7	52.36/52.29	55.52/55.45	S-SO_3	H_2S	
				1.16/1.09	0.077	Gew.-%

Analyses of spent iron-ore
Tower I

Tray	H_2O %	Sulfur content % moist	Sulfur content % dry	Loaded 1940	Shipped 1940	Designation of freight cars
1	11.3	41.50/41.51	46.79/46.80	1.11	2.11	Schwerin 23559
1+2	10.5	42.66/42.47	47.66/47.45	"	"	Deutsch 387598
2	9.0	44.10/44.13	48.46/48.49	"	"	France 261169
3	9.5	43.58/43.66	48.15/48.24	2.11	4.11	B 126728
4	8.5	44.80/44.89	48.95/49.07	"	"	Schwerin 17179
4+5	8.75	46.64/46.76	51.11/51.24	4.11	6.11	Deutsch 310291
5	10.0	40.40/40.34	44.89/44.82	"	"	Schwerin 30210
~~6~~	~~8.0~~	~~48.60/48.86~~	~~53.83/53.12~~	5.11	"	Nurnberg 15108
7	8.25	50.00/49.92	54.50/54.41	"	"	Schwerin 21567
8	13.0	42.13/42.26	48.42/48.58	6.11	7.11	Nurnberg 6103
9	13.5	41.97/41.57	43.52/48.07	"	"	
						B- 98784

Tray	H_2O %	Sulfur content % moist	Sulfur content % dry	SO_3/S %
10	10.5	44.47/44.65	49.68/49.86	0.68

Analyses of spent iron-ore
Tower 1

Tray	H_2O %	sulfur content %moist	sulfur content %dry	loaded	shipped	freight car#
1	12.5	47.30/47.30	54.06/54.06	25.6	25.6	Deutsch-382296
2	9.5	50.41/50.52	55.70/55.82	"	"	Deutsch-320101
2+3	7.5	53.81/53.82	58.17/58.18	26.6	26.6	Schwerin-12937
3	10.5	48.00/47.64	53.63/53.23	27.6	27.6	Nurnberg-11130
3+4	8.0	49.22/49.47	53.50/53.77	28.6	28.6	Schwerin-33175
4	8.25	48.13/48.54	52.46/52.90	"	"	Nurnberg-18386
5	7.75	47.40/47.60	51.38/51.59	29.6	29.6	Nurnberg-21767
5+6	8.0	48.54/48.40	52.76/52.61	"	"	Schwerin-32612
6	8.0	50.51/50.56	54.90/54.96	30.6	30.6	Nurnberg-21636
7	16.0	39.18/39.12	46.64/46.57	1.7	1.7	Nurnberg-18124
8	15.25	41.12/40.82	48.53/48.16	"	"	Nurnberg-7060
9	12.5	43.86/43.61	50.12/50.00	2.7	2.7	Schwerin-14744

Tray	H_2O %	sulfur content %moist	sulfur content %dry	$S-SO_3$
10	10.0	48.96/48.83	54.40/54.25	0.83%

Iron-determination of the Lautamass (method of the Lauta-works)

H_2O: Drying at 105°C. to a constant weight, using a weighing bottle.

Fe_2O_3: 1 gram of the dried substance is put in a 600 ccm. beaker and moistened with water. The following chemicals are added:
HCl conc. 10 ccm.
H_2SO_4 1:1 15 ccm.
HNO_3 conc. 5 ccm.

Heat on a sand bath until dry. After cooling add 20 ccm. conc. HCl and 200 ccm. hot water. Boil on the sand bath until all salts, except SiO_2 are dissolved. Add such an amount of $SnCl_2$ solution that, after the reduction is completed, 1 drop of the $SnCl_2$ is in excess. Add cold water and 10 ccm $HgCl_2$ solution (5%).

Pour approx. 3 liters water into a dish add 60 ccm. $MnSO_4$-phosphoric acid solution and so much K-MnO_4 solution that the mixture becomes a slightly red color. Pour the solution to be analysed into the water, flush the beaker and titrate by means of $KMnO_4$ $\frac{N}{10}$ until the solution becomes a slight pink color.

Consumed ccm. $KMnO_4$ $\frac{N}{10}$ x 0.8 = %Fe_2O_3 of the dry substance.

%Fe_2O_3 x 1.3385 = %$Fe(OH)_3$

CaO: 0.5 g. of the dried substance are treated as before. The solution of the salts together with the undissolved SiO_2 is oxidized by 5 drops HNO_3. The oxides are precipitated by ammonia filtered and washed. The oxides are dissolved in HCl, precipitated by ammonia, filtered and washed. The filtrated are united and the CaO is precipitated. The liquid is allowed to settle for 12 hours, the calcium oxalate is filtered, washed and dissolved in diluted H_2SO_4. The hot solution is titrated with $\frac{N}{10}$ $KMnO_4$ solution.

Consumed ccm. x 0.56 = %CaO in the dry substance.

Water Determination

Investigation of iron ore for gas purification purposes

10 g. of the iron ore is weighed in a weighing bottle and transferred into a 250 ccm. boiling flask. Add 200 ccm. xylol and extract over such a period of time until the xylol is clear. After 2 hours the extraction will be finished. The extracted water volume is read from the graduated part of the apparatus.

Calculation: $\frac{\text{ccm. x 100}}{\text{weight}}$ = % H_2O

Controletest

Weigh 5 g. in a weighing bottle and dry for 2 hours in a drying oven at 110°C. After cooling for ½ hour in the desiccator weigh again.

Calculation: $\frac{\text{difference x 100}}{\text{weight}}$ = % H_2O

Alkali-content

Quantity to be tested: 1 g.

Put the sample into an Erlenmeyer flask, add 100 ccm. H_2O and boil for 15 minutes. Cool. filter and wash the residue thoroughly with warm water. Allow the filtrate to cool and titrate with $\frac{N}{10}$ HCl.

~~Add 2 drops of phenolphtalein and titrate until the red color has~~ disappeared. Add 1 drop methyl orange and titrate to a permanent red color.

Calculation: phenolphtalein titration = ccm/kg.
methyl orange titration = ccm/kg.

Iron determination in the Lauta-ore, according to Zimmermann-Reinhardt

Quantity to be analyzed: 1 g.
Add 50 ccm concin HCl and a small quantity of potassium chlorate and boil for 15 minutes, As soon as HCl fumes are appearing the oxidation has been finished. Filter into a 250 ccm volumetric flask and dilute with water to 250 ccm. Take an aliquot of 50 ccm, bring to a boil and reduce drop by drop with a $ZnCl_2$ solution until the color has disappeared. Add 100 ccm already boiled and cooled water and after 2 minutes 10 ccm $HgCl_2$ solution whereby a white, silk-like precipitation of calomel (HgCl) will appear. (Should the precipitate be of a grey color repeat the analysis). Dilute with H_2O to 500 ccm. add 8 ccm. of $MnSO_4$ solution and titrate with $\frac{N}{10}$ $KMnO_4$ until a slight pink color appears.
pink

Calculation: $\frac{\text{Factor x ccm. x 100}}{\text{Sample}}$ = %Fe

Average sulfur-content of the spent iron-ore

	1940	1941
Boehlen	44.26 %	37.35 %
Magdeburg	45.19 %	46.29 %
Schwarzheide	39.35 %	-
Zeitz	51.53 %	33.86 %
Average	45.56 %	39.34 %

Another 71.65 tons were shipped without charge to the gas coke syndicate.

Analysis of spent iron-ore

Freight car #	H_2O %	Results % S	Analysis made by the Brabag communicated to Leonhardt Sons % S	Analysis repeated May 21, 1937
34 599	12.0	38.68/39.88	38.6	[illegible]
45 372	11.0	35.96/35.81	35.8	[illegible]
46 050	14.0	36.77/36.98	36.7	[illegible]
3 146	13.0	41.07/41.25	41.0	40.40
14 900	16.0	36.42/36.43	36.4	36.24
54 906	13.5	38.18/38.22	38.2	39.03
91 700	12.5	41.46/40.55	41.6	41.60
55 485	14.5	35.00/35.04	35.1	35.27
13 818	14.0	35.95/35.59	35.9	36.08
39 053	10.0	37.34/37.36	37.3	37.18
30 839	10.0	40.47/40.49	40.4	41.05

Quantative Analysis

of the desulphurized and water-free iron-ore from Magdeburg

Al ...	4.20 %
Fe ...	38.80
Fe ...	30.70
Ca	0.60
Mg	Trace
H_2S	2.50
SO_4	15.6
$Na_2S_2O_3$	6,2
Na_2CO_3	0.0
SiO_2	1.5

of the refreshed iron-ore from the Jakob Co. Bad Kreuznach

H_2O	48.2%
H_2S	0.0
CO_2	Trace
Cl	Trace
Na_2CO_3	0.22
SiO_2	3.26
SO_3	26.21
Fe_2O_3	24.03
Al_2O_3	8.23
CaO	4.52
MgO	1.00
SO_2	0.22
CNS	Trace

Analysis of the Lauta mass made March 8, 1939

H_2O	55.3 %	
Cl	0.38	
$NaHCO_3$	0.12	
Na_2CO_3	2.38	
H_2S	0.0	
Fe_2O_3	25.4	(Water containing) water free: 56.7 %
Bulk density	0.657 g/ccm.	

Analysis of the Ash

Fe_2O_3	62.1 %
Al_2O_3	18.0
CaO	7.5
MgO	2.0
Na_2CO_3	2.0
SO_3	0.3
SiO_2	7.6

From content of the Lautamass determined March 15, 1939

H_2O	53.5 %
Fe_2O_3	23.5 % (Boehlen-method 50.5 % free from water
Fe_2O_3	23.3 % (Lauta method 50.1 % free from water

Analyses of unused iron-ore-mass

Date of ship.	Bulk Dens. kg/l.	H_2O %	Fe_2O_3 %	water-free	$NaHCO_3$ %	Na_2CO_3 %	SiO_2 %	Al_2O_3 %	CaO %	MgO %
					1938					
3/15-18	0.755	63.5	29.0		4.11	1.48				
5/6	0.611	55.0	32.3		3.78	1.90				
5/17	0.622	57.0	32.4		2.43	2.43				
5/19	0.585	54.9	33.5		2.52	2.43				
7/16	0.578	56.5	31.8		3.27	2.06				
9/5	0.668	57.0	27.4		2.43	2.01				
9/7	0.655	59.0	29.0		2.60	2.01				
9/26	0.547	54.5	32.38		2.60	1.80				
9/30	0.489	52.0	32.38		2.44	1.80				
10/13	0.624	56.5	21.15		2.47	2.40				
10/24	0.636	56.5	21.15		2.44	2.44				
12/20-1/4	0.643	57.5	21.86		2.94	3.02				
					1939					
1/16	0.697	55.0	22.55		1.48	2.35				
3/22	0.574	58.0	22.40		3.56	2.12				
3/29-4/1	0.650	58.0	22.37		1.71	1.06				
5/25-5/31	0.622	57.25	23.17		3.62	1.17	0.77			
9/1	0.694	53.67	24.30	52.5	0.31	2.36	0.77	8.75	5.24	1.06
9/8	0.702	53.02	23.64	50.2	0.02	2.38	0.04	9.42	5.02	0.94
9/25	0.688	57.90	23.80	56.67	0.06	3.00	0.95	7.95	4.03	0.86
11/24	0.756	57.46	22.92	54.57	1.41	0.71	1.22	6.65	4.63	0.94
					1940					
5/15	0.705	54.39	26.2	57.44	1.68	2.12	-	10.6	3.86	Tracen
7/25	0.658	55.86	22.56	50.56	1.68	3.43	-	6.80	3.49	0.54
7/25	0.656	55.16	22.40	49.34	1.70	3.86	-	6.82	3.11	0.52
7/25	0.684	56.15	21.84	49.00	1.62	3.59	-	6.99	3.21	0.65
8/6	0.708	52.00	27.30	56.87	0.40 NaOH	2.02	-	12.45	2.83	Tracen
8/21	0.714	51.00	25.94	53.00	0.84	5.30	-	11.20	3.18	Tracen
9/7	0.698	53.50	23.08	49.63	NaOH -	5.44	-	7.89	3.71	1.70
11/27	0.659	52.50	26.02	54.79	0.04	0.42	2.28	8.07	7.66	1.69
					1941					
5/13	0.728	56.0	23.95	54.43	NaOH 0.72	1.81	2.70	11.90	1.70	0.72
6/20	0.664	56355	25.55	58.73	" 0.56	1.48	1.45	9.85	2.29	0.36
7/24	0.627	5650.0	21.15	46.06	" 0.52	3.39	2.04	15.55	0.88	Tracen
8/10	0.657	54.0	22.88	49.73	" 0.49	3.15	1.92	16.45	1.03	Trace
10/28	0.679	55.0	23.95	53.22	" 0.40	0.42	3.43	11.58	1.70	Trace
1938	0.6178	56.66	28.69	66.3	2.84	2.15				
1939	0.6729	56.29	23.14	51.9	1.52	1.89	1.00	8.19	4.73	0.95
1940	0.6853	53.82	~~24~~ 24.42	52.9	1.14	3.27	2.28	8.85	3.88	1.02
1941	0.671	55.50	23.50	52.9	0.54	2.05	2.31	13.07	1.52	0.54

Sulfur-balance of the Boehlen factory

Introduced with the raw materials	Tons sulfur per year
Char	6,870
Tar, light-oil	6,390
Iron-ore	125
H_2S-Gas	290
Total	13,675

Leaving with intermediate products	Tons sulfur per year
Crude water gas	3,540
Multi-clone dust, producer slag	3,330
Crude gasoline	85
S-gas	1,070
G-gas	4,210
Tank-e-water	720
Phenol-water	149
Waste-water	73
Hydrogenation residue	225
Surplus gas to A.S. W.	273
Total exit	13,675

Leaving with final products	Tons sulfur per year	
Sulfur	(4,580	
Sulfuric acid	1,450	8505
Spent iron-ore	2,185	tons/yr.
H_2S-gas to hydrogenation	290)	
Finished gasoline	36	
Hydrogenation-residue	225	
Sulfide and phenolatic liquors	149	
Surplus gas to A.S.W.	273	
Fuel gas	59	
Multiclone dust and slag to power station	2,590	
Multiclone-dust and slag to dump	323	
Finished water-gas	33	
Stack claus kiln	1,390	
Stack sulfuric acid factory	92	
Total exit	13,675	