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A.G. FUER STICKSTOFFDUENGER KNAPSACK

Hirschkind & Lowdermilk

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

~~RESTRICTED~~

AKTIEN - GESELLSCHAFT FUER STICKSTOFFDUENGER

KNAPSACK BEI KOELN, GERMANY

29, 30 June 1945

Reported by:

W. HIRSCHKIND, CWS, Hq ETOUSA
F.R. LOWDERMILK, CWS, Hq ETOUSA

26 July 1945

CIOS Target No. 22/184
Miscellansous Chemicals

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

RESTRICTED

14 p. diagrs.

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PERSONNEL OF INSPECTION TEAM

W. Hirschkind.....CWS, Hq ETOUSA
F. R. Lowdermilk...CWS, Hq ETOUSA
G. B. Panero.....CWS, Hq ETOUSA
L. C. Turnock.....CWS, Hq ETOUSA

AKTIEN - GESELLSCHAFT FUER STICKSTOFFDUENGER

KNAPSACK BEI KOELN, GERMANY

1. GENERAL.

The company is a succesor of the former "Kalkstickstoffwerke, Westerregeln" founded about 1907. It was taken over by I.G. Farben probably in the 20's and is today a fully owned subsidiary of I.G. It has only one plant which is said to be the largest single carbide manufacturing unit in Germany, but owns one subsidiary "Kalkwerk/ Stromberg/Hunsruek", a limestone quarry and lime plant.

2. ORGANIZATION.

The owners, I.G. Farben, are represented by a directorate of 3 composed of the following:

Dr. Max Bachmann, Chairman
Dr. Karl Weibezahn
Dipl. Ing. Hans Mayer

Of these 3, only Mr. Mayer resides at the plant and holds the position of General Manager. The two non-resident directors are occupied with corporate and commercial matters. The plant management consisted of, besides Mr. Mayer, the General Manager, of Dr. Robert Hegals, Assistant Manager, Dipl. Ing. Frederic W. Arnet, Works Manager, Dr. Behringer, Chief Chemist.

The original investment was RM 16,000,000; the value before bombing was estimated at RM 50,000,000.

3. BOMB DAMAGE.

Five bombing attacks in October and December, 1944, and in February, 1945, damaged the plant very severely and caused suspension of operations. The damage is estimated as follows:

Building.....47%
Machinery and equipment...34%

At the time of our visit on June 29, one carbide furnace had been restored and was operating.

4. PLANT.

a. Manufactured Products.

(1)	Calcium carbide	25,000	tons/mo
	of which was sold	5,500	" "
(2)	Calcium cyanamide	10,000	" "
	all of which was sold		
	Carbide used	7,500	"
(3)	Acetaldehyde	6,000	" "
	of which was sold	4,000	" "
	Carbide used	12,000	"
(4)	Acetic Acid	400	" "
	Acetaldehyde used	300	"
(5)	Acetic acid - acetic anhydride	1,400	" "
	Acetaldehyde used	1,000	"
(6)	Acetone	400	" "
	Acetaldehyde used	700	"
(7)	Ferrosilicon	1,000	" "
(8)	Activated charcoal	80	" "

b. Raw Materials and Services.

	Quantity/mo	Cost/unit
Power (purchased from Rhienisch-Westphalisch Electricitat Werk)	105,000,000 KWH	RM 1.2-1.7/ KWH
Steam (purchased from R.W.E. Werk)	50,000 tons	RM 2.00/ton
Water from Erfttal 5 km		RM .015/ton

c. Manufacturing Operation.

(1) Calcium carbide.

Calcium carbide was produced in 11 furnaces, 7 of the old type built between 1921 and 31, and 4 of the new type built between 1931 and 1938. The old furnaces operated at 10,000 KW and produced 60 to 70 tons carbide in 24 hours. The power characteristics were: 3 phase, 50 cycles 86 % power factor, primary voltage 600. Voltage across electrode 118-151. 4.4 KWH were required per kg carbide. The new furnaces operated at 23,000 KW and produced 150 tons carbide per day. The power characteristics were: 3 phase 50 cycles 92% power factor. Voltage across electrodes 125-181. 3.2 KWH were required per kg carbide.

Soderberg continuous electrodes were used and were prepared by mixing the following ingredients:

24 pts anthracite fines
24 pts coke "
32 pts anthracite 1" size
20 pts pitch and tar

The mixture was primed with a light steel casing octagonal shaped 1050mm in width and partly graphitized. The partly formed electrodes were then mixed into holders where the graphitizing took place by heat conduction from the furnace. The electrode travel into the furnace was 40 cm in 24 hours. The electrode consumption was 30 kg per ton carbide. The raw materials, coke and lime, of nut size, were mixed in a ratio of 68 to 100 and charged continuously into the furnace. The carbide was discharged from the furnace intermittently by tapping the furnace with iron bars used as auxiliary electrodes. The molten mass ran into a kettle, mounted on cars and holding 700 kg carbide. It solidified and cooled within 20 hours after which it was crushed. The coarse material was filled into iron drums for sale while the finer material was for home use.

Two of the new furnaces are covered for recovery of gas having the following composition:

CO.....80%
H_2.....11%
CO_2..... 9%

No chemical utilization of this gas had been attempted but it was used for drying anthracite and coke, each containing about 16% moisture.

Raw material used per ton carbide:

	Quantity	Cost
Lime	1.00ton	
Power	.4 KWH	RM .012 - .017/KWE
Coke from Ruhr	.72 tons	RM 20 - 22/ton
Anthracite from Aachen	.17 kg	RM 18 - 19/ton
Coal tar	4.00 kg	
Pitch	4.00 kg	
Iron bars for tapping	3.00 kg	
Sheet iron for drums	1.00 kg	

(2) Calcium Cyanamide.

Calcium carbide was converted into calcium cyanamide by the Polzenius process, which consisted in passing pure nitrogen over calcium carbide in presence of a catalyst. The reaction:

$CaC_2 + N_2 \rightarrow CaCN_2 + C$ is exothermal and does not require heat after starting. A temperature of 700°C was reached in the furnaces. The catalyst consisted of calcium chloride and calcium fluoride in case of the tunnel furnaces and of calcium chloride alone in case of the continuous rotary furnaces.

(a) Batch Process in Tunnel Furnace.

6 tunnel furnaces are installed, each having a capacity of 25 to 30 tons/24 hours. The carbide was finely ground, mixed with the catalyst 2% $CaCl_2$ and .5% CaF_2, filled into steel boxes, and conveyed through the furnaces on trucks. 5 kg calcium nitrate was added to each box to bring the material to reaction temperature. Nitrogen of high purity was passed through the furnace counter currently. The boxes containing cyanamide on leaving the furnace were allowed to cool, after which the content was ground, screened and packed.

(b) Continuous Process (See Sketch).

The continuous process for manufacturing cyanamide represents a decided advance in the art.

The continuous furnace, 4 of which are installed in Knapsack consists of a rotary kiln of 3m diameter and 13m length, provided with a special brick lining. Fine grinding of the carbide was unnecessary and a particle size of .3-2.3mm was satisfactory. 1.5% $CaCl_2$ was added as catalyst to the carbide, which was charged with the furnace through a feed hopper. Nitrogen of 99.5% purity passed through the furnace counter-currently. The finished cyanamide was discharged into a hopper, passed through a cooling drum and thence to a screen where all material under 4mm size was recovered and sent to a storage bin. Material over 4mm size went to a crusher, was returned to the screen and sent to the bin. The standard package was a 50kg paper bag.

(c) Product.

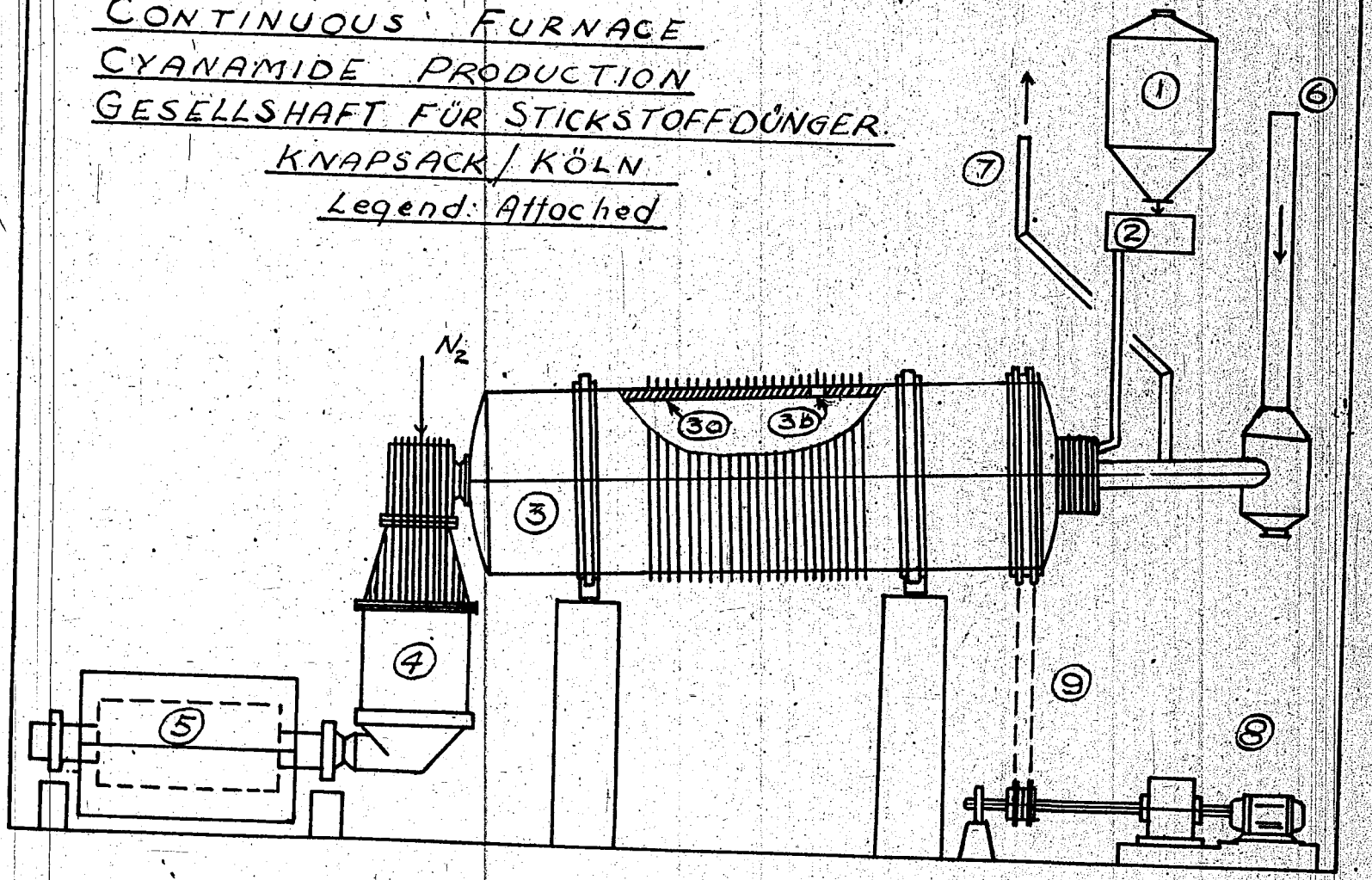
The standard product contained 22% nitrogen. The continuous process furnished a material containing 24% nitrogen which was usually reduced to the standard by adding blast furnace slag. Moreover, the cyanamide from the continuous process was non-dusting.

(d) Raw Material Used Per Ton Cyanamide.

Calcium carbide	.75 tons
Calcium chloride	20.00 kg
Calcium fluoride	15.00 kg

F.R.L. 7-24-45

CONTINUOUS FURNACE
CYANAMIDE PRODUCTION
GESELLSHAFT FÜR STICKSTOFFDÜNGER
KNAPSACK / KÖLN
Legend: Attached



LEGEND: Continuous Cyanamide Furnace.

(1) Carbide Bunker
(2) Feeding Apparatus
(3) Rotating Furnace (2.56 RPM)
(3a) Furnace lining
(3b) Pyrometer
(4) Bunker
(5) Cooling Drum
(6) Pipe for Heating up Gas
(7) Vent for normal operation
(8) Drum drive
(9) Chain drive

(3) Ferrosilicon.

Rheinisch Westphalisch Electricitat Werk once operated 4 ferrosilicon furnaces which were built in 1915. They were taken over by Gesellschaft fuer Stickstoffduenger and 2 of them were operated at 4,500 KW each. The production was approximately 1,000 tons/mo.

Raw material used per ton ferrosilicon:

Coal	.7	tons
Quartz	1.2	"
Iron borings	.7	"
Electrodes	.12	"

(4) Generation of Acetylene from Carbide.

Two methods were used for generation of acetylene.

(a) Wet Method - Carbide was dropped continuously into a large volume of water in an agitated tank. The gas was taken off from the top while the milk of lime was pumped out of the bottom and discarded. Capacity of 2-2.5 tons carbide/hr.

(b) Dry Method - The generator used for this method had the principle of a Herreshoff Furnace, with 6-8 revolving hearths. Carbide and water were fed on the top hearth while dry calcium hydrate left the bottom hearth. It was mixed with coal dust, pressed into briquettes and calcined in a shaft kiln. It yielded a very active lime which is recycled to the carbide furnace.

Capacity of generator - 4 tons carbide/hr.
Capacity of shaft kiln for lime - 90 tons/day.

(c) The acetylene from both type generators went to a gas holder, a meter and to the following purification steps:

1 Tower where the gas was washed with chlorine water containing 1 gpl Cl_2

2 Towers in series for washing with 4% caustic soda solution. Dimensions of all towers - 2m diameter. 10m height.

1 Short tower of same diameter for water removal. Capacity of purification system - 6000 cbm acetylene/hr.

(5) Acetaldehyde.

The installation consisted of 6 generators of 1m diameter and 13m height, rubber or brick lined and filled with liquor of the following composition:

10% $FeSO_4 \cdot Fe_2 (SO_4)_3$ together with pure $Fe_2 (SO_4)_3$.

The solution was maintained at 50% in ferric and 50% in ferrous condition by partial withdrawal and oxidation with air. The SO_4 and catalyst content was as follows:

30% SO_4 (total)

1% Hg (as Hg SO_4)

Acetylene was passed through the tower at a rate of 1400 cbm/hr of which 800-1000 cbm are absorbed. The exit gas containing a mixture of acetaldehyde and acetylene was stripped by passing through a tubular cooler (gas in tubes, water in shell) thence through a wash tower and to waste.

The product consisting of crude aldehyde as 10% aqueous solution was continuously distilled in 4 columns.

(a) 1 column 2m diameter, 6m height, packed with Raschig rings. 70% aldehyde distilled over.

(b) 2 columns - first: 2m diameter, 10m height packed with Raschig rings. Second: 2m diameter 12 trays. Heating coils of iron. Overhead product 99.2% aldehyde.

(c) Final column - 8m diameter, 6m height, for stripping acetylene.

All distillations were carried out at 1 atm gauge. Production - 6,000 tons/month.

(6) Acetic Acid, Acetic Anhydride.

Approximately 1300 tons of the aldehyde were converted into to acetic acid and acetic anhydride. Two processes were used:

(a) Old System - manganese process for production of acetic acid only. Aldehyde at a rate of 700 liters/hour mixed with oxygen was fed to an aluminum lined tower, 6m diameter by 10m height. Catalyst was manganese acetate.

(b) A new system was a batch process for simultaneous production of acetic acid and acetic anhydride, using cobalt acetate and copper acetate as a catalyst. A cylindrical vessel of 6 cbm content equipped with agitator and cooling coils was filled with 700 liter acetic acid containing the catalyst - 10kg cobalt acetate and 6 kg copper carbonate. 1600 liter aldehyde was added to the catalyst mixture heated to 40°C while oxygen was passed through for 4-5 hours.

Crude product:		
	Acetic anhydride	- 33%
	Water	10%
	Acetic Acid	57%

The crude product was purified by vacuum distillation:

1st Step - Water and pure acetic acid were driven over in a column of 12m height, 1m diameter, built of high silica iron (Duriron). The bottom consisted of a mixture of 40-42% acetic anhydride, acetic acid plus catalyst.

2nd Step - The overhead of pure acetic acid and water was concentrated by vacuum distillation to 95% strength and used for manufacture of acetone.

3rd Step - The remaining mixture of 40-42% acetic anhydride, acetic acid and catalyst was freed from the latter by vacuum distillation. It is then ready for sale or conversion into acetic acid or vinegar.

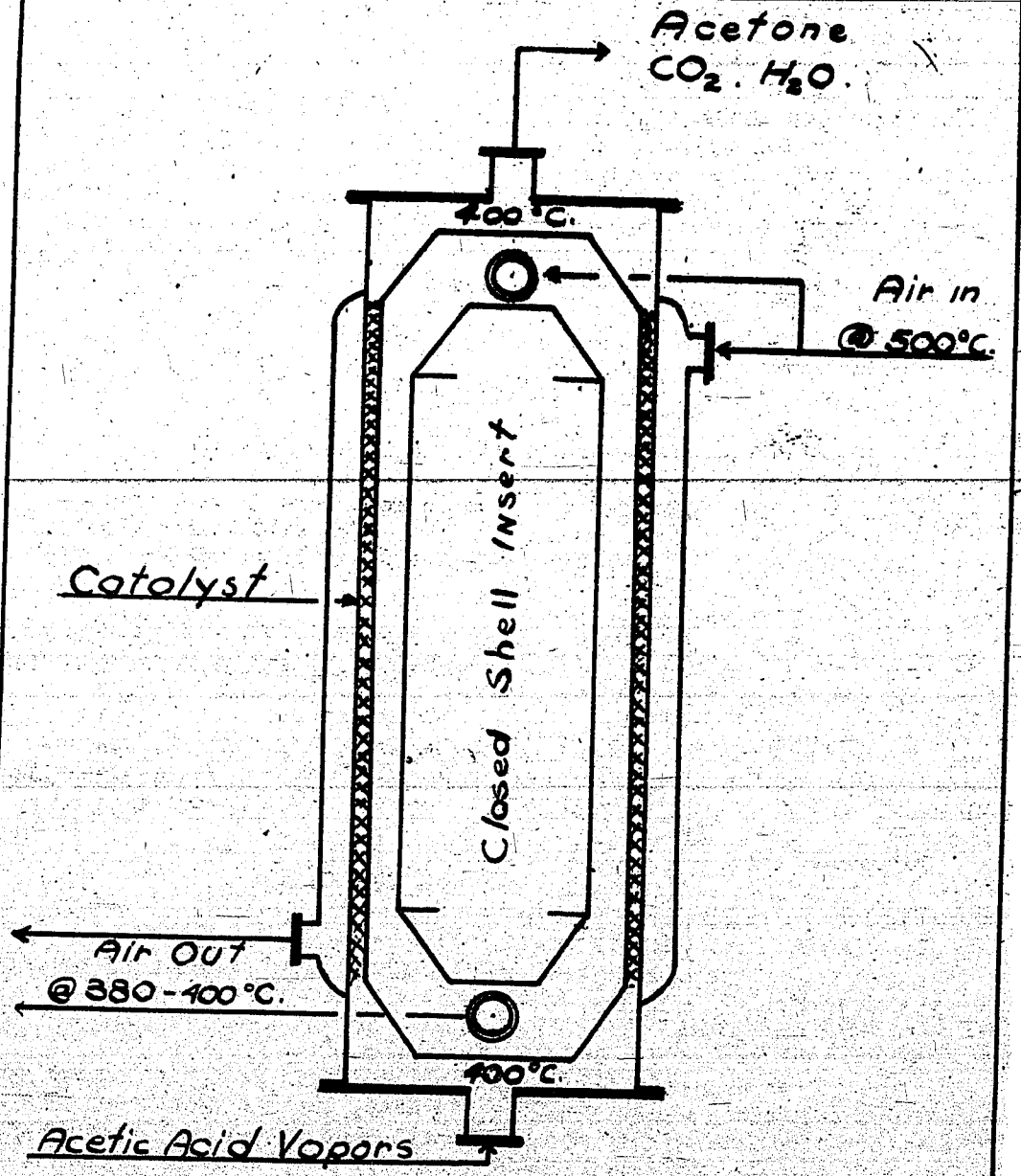
Stills for steps 2 and 3 are same as for Step 1.

Production: Acetic acid - 400 tons/mo
Acetic acid acetic anhydride mixture 1400 tons/mo.

(7) Acetone.

Acetic acid (95%) was vaporized and the vapors heated to 400°C in a tubular 4-pass gas fired furnace, through which combustion gases are circulated prior to their passing on to the heating zone of the reactor. The preheated acetic acid vapors were passed to an annular type reactor in which they were converted, at 400-420°C, to acetone by means of cerium acetate catalyst.

The reactor (see sketch) was of annular type with the catalyst packed in the annulus, around which the combustion gases were circulated.



ACETONE REACTOR

GESELLSCHAFT FÜR STICKSTOFFDÜNGER

KNAPSACK/KÖLN

F.R.C.
7-19-48.

Catalyst : 10-15mm size pumice particles were impregnated with a solution containing 100kg acetic acid and 100kg cerium acetate in 500 liter water and dried to a cerium oxide content of 10%.

The annular reactors had a concentric space of 10mm for catalyst chamber regardless of length or diameter. Five reactors of the following sizes are installed:

(a)	1300 liters	catalyst volume -	production of acetone 200 liter/hr
(b)	2500 "	" "	" acetone 400 liter/hr
(c)	1500 "	" "	" acetone 200 liter/hr
(d)	1500 "	" "	" acetone 200 liter/hr
(e)	2000 "	" "	" acetone 300 liter/hr

Capacity of all 5 reactions: 800 tons/month. Coke oven gas was used for firing (4000 cal/cbm).

Distillation: The crude acetone was distilled continuously in 2 steel plate columns, the first one with 48, the second with 60 plates.

Yield: 100% of theory.

Materials of Construction:

Kettles, coils and pipe for handling acetic acid liquid and vapors up to entering the heating furnace - aluminum.

Tubes in furnace for heating acetic acid vapors - stainless steel.

Reactor and distillation equipment - steel.

Raw materials per ton acetaldehyde, acetic acid, acetic anhydride, acetone:

Nitric acid 100%.....	23.	kg
Sulfuric acid 100%.....	14.	kg
Ferrous sulfate.....	23.	kg
Mercury.....	2.3	kg
Caustic soda.....	32.	kg
Cupric carbonate.....	.045	kg
Cobalt acetate.....	.045	kg
Potassium permanganate.....	.090	kg
Pumice.....	.7	kg
Cerium carbonate.....	.35	kg

(8) Activated Charcoal.

Approximately 150 tons of activated charcoal were recovered per month as by-product. 150 tons hydrochloric acid were used in this recovery.

MONTHLY AVERAGE PRODUCTION

	Carbide	Calcium-Cyanamide	Ferrosilicon	Acetaldehyde	Acetic Acid	Acetone	Acetic Acid Anhydride	Activated Charcoal
	t	t	t	t	t	t	t	t
1930	13 140	7 700	-	1 855	975	109	-	47
1931	8 595	4 265	357	1 618	1 003	85	96	83
1932	8 756	5 975	237	1 360	754	69	170	43
1933	10 624	6 515	344	1 740	1 079	151	279	50
1934	14 076	8 470	1 017	2 126	1 173	206	324	69
1935	17 431	9 650	708	2 865	1 535	290	422	83
1936	17 704	10 190	761	3 243	1 593	325	641	81
1937	19 524	8 800	2 007	4 415	1 669	446	817	72
1938	20 172	8 900	2 000	4 407	1 593	495	912	61
1939	20 705	9 260	2 140	4 214	1 513	696	1 002	91
1940	21 995	7 105	1 725	5 021	1 717	548	1 062	84
1941	24 501	6 425	569	6 086	2 073	594	1 151	82
1942	23 784	3 660	445	6 240	2 080	635	1 037	81
1943	22 777	2 550	963	6 081	2 048	666	954	86
1944	16 204	1 250	677	3 880	1 426	499	517	27
1945	1 500	1 800	-	-	-	-	-	-