

(Interrogation of Dr. Leo Schlecht)

**CARBONYL NICKEL AND
CARBONYL IRON POWDERS,
THEIR
PRODUCTION AND PROPERTIES**

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

Price 3s. 6d. net.

S.O. Code No. 51—5275—75

CARBONYL NICKEL AND CARBONYL IRON POWDERS,
THEIR PRODUCTION AND PROPERTIES.

Composite Report of Interrogations of

DR. LEO SCHLECHT

Badische Anilin und Sodafabrik
(formerly I.G. FARBEN, GEFPAU)

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Bios Target No: C21/622.

C.I.O.S. Black List Item No.21.
METALLURGY

British Intelligence Objectives Sub-Committee,
32 Bryanston Square, London, W.1.

INTERROGATION OF

DR. LEO SCHLECHT OF BADISCHE ANILIN UND SODAFABRIK

(FORMERLY I.G. FARBEN, OPPAU)

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INTERROGATION OF

DR. LEO SCHLECHT OF BADISCHE ANILIN UND SODAFABRIK

(FORMERLY I.G. FARBEN, OPPAU)

GENERAL NOTES

PERSONAL HISTORY

Born in 1897, Dr. Schlecht studied chemical engineering at the Technische Hochschule at Stuttgart, obtaining his degree of Dr. Ing. in 1922 after carrying out research work on the electrochemical behaviour of chromium. He was a lecturer at the Technische Hochschule for six months after graduation and subsequently joined I.G. Farben at Oppau, where he has remained ever since, having been absorbed by the new organisation which is now controlling the works of I.G. Ludwigshafen and I.G. Oppau.

Dr. Schlecht was initially engaged on examining the synthetic manufacture of petrol, and in 1923 it had been noted that iron carbonyl had shown promising results as an anti-knock. In 1924 he designed a plant for the production of liquid iron carbonyl on a production basis and later found that iron powder produced from its decomposition possessed special electro-magnetic properties.

Dr. Schlecht's experience with iron carbonyl, availability at Oppau of high pressure equipment, mechanics skilled with this apparatus and suitable workshops, led him to investigate the practicability of producing nickel carbonyl under pressure from nickel-copper matte.

In 1937 he was appointed a "Prokurist" of I.G. Farben, which entails powers of attorney with regard to matters concerning production and development of nickel and carbonyl iron, but excluding sales matters, this appointment conferring signature powers. The salary appropriate to this appointment was understood to be of the same order as that of "Direktor". It was stated that Dr. Schlecht was offered the appointment of Direktor on several occasions since 1937 but refused, feeling that his interests lay in technical development rather than administration.

As the leading German nickel production specialist, Dr. Schlecht was employed in mobilising his country's nickel resources and refining capacity. He visited Palermo in 1940,

arranging for the completion of mining and smelting equipment. He negotiated the various arrangements with several smelters and refineries in Germany, Norway and Belgium, in order to build up German production to 8860 metric tons of nickel from Petsamo ore during 1944. At the same time he remained in charge of the Oppau nickel refinery and carbonyl iron production.

PRESENT APPOINTMENT

Following the splitting up of I.G. Farben after the German surrender, an organisation under the name of Badische Anilin und Sodafabrik took over all the assets, equipment and administration of the I.G. Farben works at Ludwigshafen and Oppau. The senior personnel of this organisation are mainly French officials with one prewar I.G. Farben director. Dr. Schlecht has remained in charge of the metals (production and research) department of this new organisation and has retained his appointment as Prokurist. The organisation and personnel of this new company are shown in the chart, Table I.

BADISCHE SODA UND ANILIN FABRIK

Mr. Fougier (French) - Head of French administration of the firm at Baden-Baden

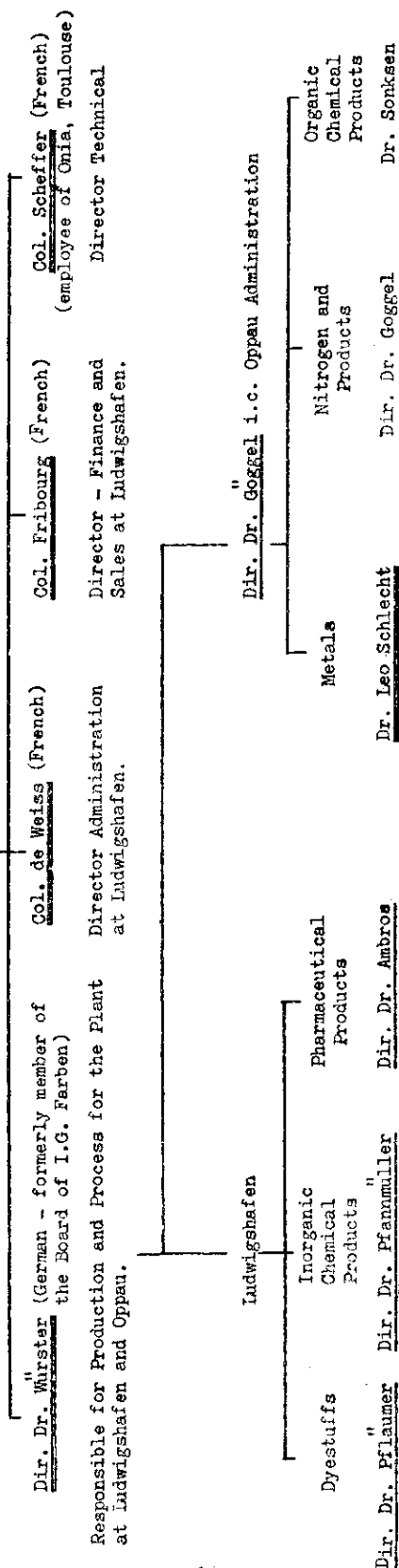


Table 1.

NICKEL PRODUCTION

INTRODUCTION

The interest in nickel powder production at Oppau followed on the successful development, manufacture and marketing of iron carbonyl initiated as an "anti-knock" for petrol, and subsequently the carbonyl iron powder production, owing to its advantageous electromagnetic properties.

During 1929, means of producing nickel carbonyl under pressure from nickel sulphide were investigated, and, following on further laboratory tests, it was established that it would be produced directly from nickel matte without the necessity for removing the major proportion of the copper by dividing into tops and bottoms. A plant was ready for commercial production in 1933.

This rapid evolution from laboratory to the production stage was facilitated by the high pressure equipment of synthetic ammonia and iron carbonyl, the availability of workshops and mechanics skilled in high pressure equipment.

Initially, metallic nickel powder was produced from nickel containing scrap and other nickel containing products, which, however, were available on the market in only limited quantities. I.G. attempted to arrange for a continuous supply of raw material and initiated unsuccessful negotiations in attempts to obtain nickel containing ores from Tasmania, Evje in Norway, South Africa, Brazil, Finland and Greece. Attempts to use ore from the Black Forest in Germany were also inconclusive owing to the small quantities available and their low nickel content.

Eventually nickel matte was obtained from Canada, with the result that the following quantities of metallic nickel were refined at Oppau.

1933	106 tons.
1934	454 "
1935	1,452 "
1936	2,652 "
1937	3,048 "
1938	4,098 "
1939	4,170 "

Other nickel production in Germany during 1933 is reported to have been as follows:-

Krupp, Frankenstein	approx.	1000 tons ore from Greece
Sachs Blaufarbenwerke		
Oberschlema	"	800 " nickel speiss from Burma
Nord Deutsche Affinerie,		
Hamburg	"	250 " nickel slag from the copper refinery.
Vereingte Deutsche Metall-	"	2100 " nickel oxide.
werke, Altena.		

PRE-WAR POSITION

The total production of nickel in Germany and Austria during the period 1934-1939, including imports, is reported to have been as follows:-

Production and Imports of Nickel Germany and Austria

Approximate Production	I n M e t r i c T o n s					
	1934	1935	1936	1937	1938	1939
I.G. Oppau	348	1435	2642	3048	4098	4170
Krupp Frankenstein	1200	800	400	465	400	400
S.B.F.W. Oberschlema	950	1200	1300	1100	1000	1000
Nord Deut. Affinerie	250	280	250	100	100	100
Altena	1475	1243	1036	1537	1500	1000
Misc. other sources	150	150	180	180	180	180
Total Production	4373	5108	5808	6430	7278	6850
Imports - Germany	3381	3647	2331	2266	2000	1000
- Austria	681	1011	1341	2276	1000	-
	8435	9766	9480	10972	10278	7850
Imports to Germany - Austria originated as follows:-						
From Inco	2601	3887	2375	3752		
From Christiansand	859	771	1297	790		

NICKEL IN GERMANY DURING THE WAR

Dr. Schlecht was appointed to make all arrangements necessary on behalf of I.G. towards exploiting the nickel mines and smelting plant at Petsamo. He visited Finland in July 1940, accompanied by an I.G. mining expert (Herr Reddehaase) and a smelting expert (Dr. Schubart), and obtained detailed information regarding the ore, the existing plant, the designs of plant and plans for completing the installations. The smelting plant was found to be only 70% complete and damage caused by the Russians in the first Finnish war had been confined mainly to the living quarters. Work was put in hand immediately to complete the equipment with the aid of the Finns, and I.G. spent some 30 million Reichmarks in getting production under way. Production started in 1942.

The output of the mine was substantially greater than that which could be dealt with by the smelter. The richest grade of ore containing $4\frac{1}{5}\%$ nickel was, therefore, sent to the Nord Deutscher Affinerie at Hamburg. Some ore was also sent to the smelter at Evje, near Christiansand, Norway.

Further ore was taken from another deposit at Nivala, S.W. of Petsamo. This was concentrated by flotation on site, by the largest copper refining company in Finland and was then sold to I.G. who sent it to the Nord Deutscher Affinerie, Hamburg. The matte produced at Hamburg from Petsamo and Nivala ores contained nickel 50/55%, copper 35/40%.

Ore for smelting was also sent from Finland to:-

Sachsische Blaufarbenwerke - Aue - Saxony
Mansfeld A.G., Sondra, near Kassel
Societe Metallurgique Hoboken, near Antwerp.

None of these three works however, produced matte on a production basis. At Blaufarbenwerke, where there was an electrolytic refinery, attempts were made to smelt 3000 tons of ore, but without success. This works had eventually to depend on supplies of matte from Hamburg for its nickel refinery. With regard to Hoboken, the Belgians were apparently very slow in getting under way and succeeded in obtaining several financial advances in payment or modifications to the plant stated to be necessary and eventually only smelted down a total of 5000 tons of ore until the end of the war, in lieu of producing the 2000 tons of nickel

in matte per annum which had been planned for them. They had been supplied with some 30,000 tons of ore in all, and Dr. Schlecht is of the opinion that this was still held by them at the end of the war.

The smelter at Mansfeld had only been intended for use if the N.D.A. were put out of action by air attack or other causes. Only some 8000 tons of ore in all were sent there, and smelting operations were only initiated towards the end of the war.

With regard to Petsamo, production started in 1942 during the second Finnish war, when German troops moved in to aid Finland. The nickel content of the ore which was processed in 1943 was about 2000 tons. This rose to 10,000 tons of nickel in matte in 1944.

Refining of matte produced from Petsamo ore, both intended and achieved, is set out below in metric tons of metallic nickel, as reported by Dr. Schlecht:-

	<u>Planned p.a.</u>	<u>Achieved in 1944</u>
I.G. Oppau	5000 metric tons.	2986 metric tons.
Blaufarbenwerke	2400 ? " "	1500 metric tons (cathodes)
Christiansand		2000 " " "
I.G. Frose	1500 " "	360 " " "
		<hr/> 6846

It is understood that a further 1200 tons of nickel per annum were obtained from the Frankenstein ores, mostly in the form of nickel iron, rather than pure nickel, by the Renn process. Small amounts of ore of Greek origin were used with the Frankenstein ores, but Dr. Schlecht had no idea of what quantities were involved, but stated that Greek ore was entirely suitable for use at that plant. It therefore appears that at the peak of German nickel production in 1944, the total metallic nickel refined was of the order of 11,000 metric tons.

NICKEL STOCKS IN EUROPE, MAY 1945.

At the time of the German surrender, appreciable stocks of nickel containing ore, matte, etc., were in transit or held in different parts of the Continent, representing the pipeline

of wartime supply from the mines in Finland through smelters and refineries to suppliers. Indications of the approximate quantities and locations of these stocks are given in the following table.

Stocks of Nickel Containing Ore, Matte etc. May 1945.

Figures received by I.G. after the end of the war

	<u>Ore</u> <u>(Tons)</u>	<u>Nickel Contained</u> <u>(Tons)</u>
<u>Ore</u>		
Kirkenes, Norway	6329	220
Hoboken	27802	970
Sondra (1)	50	1.5
Blaufarbenwerke	2926	122
Schoenebeck (Elbe) (2)	1906	66
Hamburg	Nil	Nil

Nickel Ore in Form of Sintered Agglomerates

	<u>Sintered Ore</u>	
Sondra	850	51

Nickel Ore/Matte Containing Iron

	<u>Ore/Matte</u>	
Sondra	150	18
Hettstett (Harz)	841	100

Nickel-Rich Converter Slag from Petsamo

	(slag)	
Froese	76	27

Nickel Matte

	(Matte)	
(a) from Petsamo smelter:-		
Christiansand S	640	320
Hamburg	467	357
Schriesheim (near Heidelberg)	550	275
(b) From Hamburg smelter:-		
Hamburg	226	98
Bremen (Nord Deutscher Hutte) (3)	123	60
Schoenebeck (Elbe) (4)	472	255
Blaufarbenwerke (at Oberschlema)	275	119
Froese	265	128
Schrieschaim	449	220
Oppau (lost under debris)	100	50

<u>Metallic Nickel</u>	<u>Ore</u> <u>(Tons)</u>	<u>Nickel Contained</u> <u>(Tons)</u>
Oppau		Nil
Christiansand S		178
Froese		176

- (1) I.G. records indicate that 5000 tons should be held at Sondra. U.S. authorities have said plant is in badly neglected condition.
- (2) En route to Sondra.
- (3) This was for an experiment involving the direct addition of matte to steel. Instructions issued by Ministry at Berlin. No success was achieved.
- (4) Partly for Froese and Blaufarbenwerke.

GERMAN WARTIME REFINING COSTS

The full control of and responsibility for the operations involving the mining and smelting, etc., of the Petsamo and Nivala ores were, according to Dr. Schlecht, held by I.G. Farben. The selling price of metallic nickel was fixed by the German Government and maintained at the prewar price of 2.45 R.M. per kilo. I.G. had to pay for production costs in advance and then recover, after months of delay, the extra over-and-above prewar costs from the German Government. None of the other firms involved had dealings direct with Berlin; they all had agreements with I.G.

Throughout the war, I.G. paid the following prices:-

For Petsamo ore - untreated	3.5 R.M./Kg. of nickel in ore
" Petsamo matte	4.0 " " " " matte
" Hamburg matte from Finnish ore	6.0 " " " "

PRECIOUS METALS FROM FINNISH ORES

During the war, the precious metal concentrates were sent by I.G. to the N.D.A. at Hamburg. They were in the copper sulphide, after which the P.M. slimes were treated for the extraction of the metals.

According to Dr. Schlecht, one ton of matte produced at Petsamo, with a 50% nickel content, contained on an average

3	gms.	gold
150	"	silver
4	"	platinum
4.5	"	palladium
0.5	"	other precious metals.

I.G. were allowed to keep sufficient of the precious metals extracted for their own industrial needs (e.g. catalysts for the oxidation of ammonia, etc.); the remainder being sold to the Precious Metal Department of the State which issued them to Heraeus, Siebert, etc. for working up.

DEVELOPMENT OF NICKEL POWDER

USES FOR NICKEL POWDER

Introduction

Only very limited development and research work had been carried out since 1933 when Oppau initiated refining nickel, owing to the severe restrictions on the use of nickel brought into force in Germany under the new Nazi economy. The most noteworthy development was the production and sintering of nickel powder for the production of sintered nickel alkaline accumulator plates, described later.

Investigations at Oppau were otherwise carried out on the following subjects: - production and use of porous nickel filters for concentrated alkali; porous nickel bearings; production and utilisation of non-porous sintered nickel powder; radio valves, nozzles for artificial silk manufacture, nickel clad steel, anodes for nickel plating, nickel-iron alloys with special magnetic properties, nickel welding wires and coatings for arc welding electrodes.

NICKEL FOR THE GERMAN STEEL INDUSTRY

Approximately 98% of the nickel powder produced by I.G. Farben was supplied to the German iron and steel industry; the analysis of this powder conformed to the following

<u>C</u>	<u>Fe</u>	<u>S</u>
0.25 max	0.7 max	0.01

It was packed into 100 or 150 Kg. steel drums, both container and powder being dropped into the furnace.

The use of carbonyl nickel powder in the melting practice of the German iron and steel industry has been published in Stahl und Eisen 1940, LX, pages 226-227. Dr. Schlecht had no information to add to this paper, written by his colleague P. Assmann, and himself.

NON-FERROUS METALS INDUSTRY.

Only a small proportion of the nickel produced by I.G. Farben was used in the non-ferrous metals industry owing to the strict German nickel conservancy measures. An article by Assmann and Schlecht on "Carbonyl Nickel Powder in the Melting Practice of the German Non-Ferrous Metal Industry", published in Metallwirtschaft, 1939, XVIII, pages 467-468, summarises Dr. Schlecht's views on the matter. Dr. Schlecht was not aware of any new developments which had been introduced into Germany during the war. The main purpose of his paper was to show that carbonyl nickel powder is as satisfactory for use in non-ferrous metal melting as any other commercial nickel available.

The nickel normally provided to the non-ferrous metal industry was similar to that described above and supplied to the steel industry. Where very special alloys were required to be made, a better quality - made by using NiCO_4 of low iron content - was supplied, with analysis approximately as follows:-

<u>C</u>	<u>Fe</u>	<u>S</u>
0.1	0.08	0.001

SINTERED NICKEL FOR NICKEL CLAD PLATES

Sintering of slabs for nickel clad steel was developed as an alternative in order to obviate the melting and rolling in normal practice. Few advantages for this method of manufacture were claimed, except Dr. Schlecht stated that slabs up to 1600 Kg. in weight could be produced by this means against the considerably smaller ingots which could otherwise be produced in Germany.

Details of the use of carbonyl nickel powder in this field are given in a publication by Dr. Schlecht and his colleague G. Trageser, which appeared in Metallwirtschaft, 1940, XIX, No.4. pages 4-6. Dr. Schlecht had nothing to add to the information contained in this paper.

SINTERED NICKEL FILTERS FOR CONCENTRATED ALKALI

A publication by Dr. Schlecht and Dr. G. Trageser on "Sintered Nickel Filters for Cleaning Concentrated Alkali" was published in Die Chemische Fabrik, 1939, XII, pages 243-244. This article describes the advantages of these filters.

A limited number of filters have been supplied mainly to the I.G. factory at Hoechst for filtering alkaline liquids. The filters were made by tamping 3-5 micron powder of 1.0-2.0 packing density with or without a proportion of filler, into moulds and sintering in hydrogen, first at low, and finally at high temperatures. The filters were prepared in the form of plates up to 30 cm. square in area, tubes up to 15 cm. in diameter and about 45 cm. in length, and cones suitable for welding to the ends of the tubes as closures.

Dr. Schlecht explained that optimum porosities and thicknesses had not been investigated by him, but he had found that the porosities of filters should be as large as possible in order to avoid clogging by the precipitant. It was considered that a coarse powder (10 microns) would be specially suitable for the preparation of filters, since

- (a) it would contract very little during sintering, and
- (b) the sintered product would contain coarse pores.

There had apparently been insufficient time to develop a powder of this type on a commercial scale at Oppau. Dr. Schlecht explained however, that coarse pores could also be obtained by the introduction of a volatile or leachable filler of suitable particle size (ammonium carbonate and urea had proved useful experimentally).

EFFECT OF IMPURITIES

No study had been made of the effect of carbon content on nickel powders. I.G. considered it essential, however, that the sulphur content should be extremely low since it was not practicable on economic grounds to desulphurise nickel powder by prolonged treatment with hydrogen during sintering. In Dr. Schlecht's view, the importance of sulphur lay solely in the fact that it could readily cause brittleness in the sintered product.

It had been noticed, however, that iron had an effect on the shape of the particles. If the iron content of a nickel powder

exceeded about 1%, the particles were spherical and possessed the onion-like structure associated with particles of iron, whereas substantially sulphur-free nickel consisted of polygonal and multicrystalline particles.

SINTERED NICKEL ALKALINE ACCUMULATOR FLATES

GENERAL

Shortly after 1933 when Dr. Schlecht had initiated production of nickel powder from Canadian matte, he commenced research and development work with a view to finding further fields in order to increase nickel consumption. The most important result of his development work was the production of sintered nickel accumulator plates with a high degree of porosity from powder of light packing density. The information he was able to give was based on the experimental work which he had carried out up to a stage when Akkumulatoren Fabrik Hanover A.G. (normally referred to as A.F.A.) were sufficiently interested to acquire the sole rights of the I.G. patents in Germany. A.F.A. subsequently undertook all further development work, details of which they did not communicate to Dr. Schlecht.

The method of sintering described below is thus only based on the laboratory tests by Dr. Schlecht, carried out at Oppau in 1938 when he discontinued development work. He does not claim that his investigations were comprehensive, but merely that they were sufficient to create a novel market for his nickel powder production. It is of interest to note that his patents were sold to A.F.A. on the payment of a lump sum of 300,000 R.M. and an annual royalty of a further 20,000 R.M.

In 1940, I.G. Oppau was selling some three tons per month of the special light packed powder to A.F.A., and this was the normal supply throughout the war. Towards the end of 1944 Oppau was asked by A.F.A. if they could increase their monthly supply to twenty tons, but I.G. were unable to conform because of bomb damage, transport difficulties, etc., associated both with their own and the A.F.A. organisation.

The sintered nickel accumulator plates were used in the main for starter batteries for German Air Force 'planes, their main advantage being low internal electrical resistance, permitting a high rate of discharge under normal service conditions. Dr. Schlecht is of the opinion that nickel cadmium alkaline batteries have no disadvantages which would prevent them from being used for motor cars and other purposes, once accumulator manufacturers are prepared to make the necessary capital outlay and convert their present production system.

PREPARATION OF THE NICKEL POWDER.

Decomposer. During the course of experimental work at Oppau, it had been found that powder of very low packing density could be obtained by increasing the through put in the standard decomposer and without unduly clogging the extraction apparatus.

The Particle Size of powder formed under normal conditions was of the order of 3-5 microns, but at the higher rate of production a proportion of the particles coalesced to form groups as large as 50-100 microns in dimension. After removal of this powder from the decomposer, it was screened by brushing it mechanically through a stationary coarse sieve, provided with holes of about 1 mm. diameter (the screens employed for screening normal nickel or iron powders contain about 180 mesh per inch).

The Packing Density of the powder as produced was 0.7 gram/c.cm. The density increased during transport by rail to 0.8 gram/c.cm. which was the minimum specified by A.F.A. Motor transport was stated to break down the agglomerates, causing an excessive increase in packing density, and was thus never used.

The packing density value of 0.8 was apparently of no special significance, but was sufficient to provide a reasonably high porosity in the sintered product. It was hoped at one time to increase the porosity by producing an even lighter powder by increasing the temperature of decomposition, but it was found that, as in the case of unagglomerated powders of small particle size, the product was very bulky and could not be handled conveniently by the normal worm gearing by which it was removed from the decomposer. Conversion of the outlet at the bottom of a decomposer was planned and commenced, but this was not completed before production ceased.

With the exception of a few tons of a finer powder sent to A.F.A. early in 1938, all supplies were of the type with 0.8 gram/c.cm.

packing density described above, and as far as Dr. Schlecht was aware, A.F.A. employed only this standard powder in their manufacture of accumulator plates.

Control Tests. The product was controlled by periodical batch analysis and determinations of the packing densities. No other tests were applied.

PRODUCTION OF THE POSITIVE PLATES

Filling the Former. The procedure adopted in the Oppau laboratories for the production of plates was as follows. The powder was fed by sieving, or by gently pouring, without any heat treatment, into an iron or steel former resting on an iron plate, and the excess was scraped away by a knife edge (without any special precautions being taken in regard to the angle of incidence of the knife blade). If desired, a wire mesh grid could be introduced. The plate and former were tapped lightly in order to smooth and slightly consolidate the powder.

A steel plate which differed in thickness in various experiments from about 1 mm. to 5 mm., was placed on the powder and lightly pressed down by hand (the weight applied being less than 1 Kg.). The dimensions of the iron plate were such that a ridge of powder approximately 1 mm. in width, remained uncompressed and slightly "proud" around the edge of the plate. The ridge served to lock the edges of the compact during sintering, thus preventing contraction in a direction parallel to the faces of the sintered plate, all contraction taking place in a vertical direction. Adhesion between the compact and the iron plates and former was prevented by painting the latter with a slurry of magnesia before use.

Sintering The complete assembly of upper and lower plates, former and powder, was introduced into a steel box in a furnace into which was passed an atmosphere of purified hydrogen. Sintering was effected by raising the temperature of the box to 1100 °C in a period of twelve hours, maintaining that temperature for two hours and allowing the box to cool during a subsequent period of ten hours, thus completing a twenty-four hour cycle.

The Density of the nickel was, however, admitted to increase from 0.8 gms./ccm. to 1.8-2.0 gms./ccm. during the preparation of the plate. This would indicate that the linear contraction, if it took place only in a vertical direction, would be 56%, and if it were assumed that it took place equally in all directions it would be about 17%. These figures are thought to be remarkably high in view of the reported absence of cracks.

The Porosity of the plates produced experimentally by I.G. Oppau was between 80 and 84%. Dr. Schlecht considered that it might well be possible to increase the porosity even to 90%. He had prepared specimens of 94-95% porosity by the use of fillers, but admitted that these plates were easily indented and that they were mechanically weak. The view was expressed that in order to extend the field of sintered accumulator plates, it is highly desirable on economic grounds to reduce the weight of metallic nickel in the plates by increasing the porosity to the maximum obtainable; this would also have the effect of increasing the ratio of the weight of active mass to nickel.

Flaking No troubles due to short circuiting of cells by particles or layers flaking from the surface of the plates had been encountered, according to Dr. Schlecht.

PRODUCTION OF THE NEGATIVE PLATES

Negative plates were first prepared by I.G. on an experimental basis from sintered iron, but these were found to disintegrate rapidly by electrical action during service. Patents for the production of nickel iron alloy plates or for the formation of nickel iron grids on iron plates, were designed, but did not prove successful in overcoming this trouble.

Dr. Schlecht did not know whether A.F.A. had introduced copper into their plates. A pure nickel plate, in his view, would have been too expensive, and in any case would not have been permitted in Germany owing to the strict nickel conservancy policy. As far as he knew, copper offered no advantage from the electrical standpoint.

TESTING

Particle size measurements were not regularly carried out on nickel powders. The Electron microscope had not been employed, nor had micro-hardness measurements been made.

The strength of the sintered plates was only judged by bending them manually. The porosity was determined from the apparent density and there was no attempt to measure the permeability. Attention was mainly given to the electrical capacity of the finished plates.

CARBONYL IRON POWDER

Introduction

According to Dr. Schlecht, research work on iron carbonyl had been initiated at I.G. Oppau, in connection with their work on the synthesis of motor fuels, when it was found that $\text{Fe}(\text{CO})_5$ diminished the knocking tendencies of petrol. Universal use of lead tetra ethyl in 1926 checked the use of $\text{Fe}(\text{CO})_5$ for this purpose, and I.G. Oppau therefore decided to produce carbonyl iron powder which had been found meantime to possess favourable electro-magnetic properties.

Very gradually this powder gained favour in the telephone and wireless industries, as will be seen from the annual sales figures.

Sales from Oppau of Iron Powder (Short tons)

1927	53	1937	540
1928	86	1938	707
1929	149	1939	1062
1930	172	1940	987
1931	105	1941	1634
1932	51	1942	1506
1933	146	1943	952
1934	256	1944	966
1935	365	1945	-
1936	486	1946*	150

* 10 months.

Method of Production.

Iron carbonyl is produced through treating iron ore or iron scrap containing 1.5% sulphur with carbon monoxide under a pressure of 200 atmospheres, the temperature being maintained at 240°C . The iron carbonyl thus formed is separated from the surplus CO during cooling to normal temperatures, and is obtained in liquid form.

The iron carbonyl whose boiling point is 103°C is then evaporated under normal pressure and decomposed into metallic iron powder and carbon monoxide in cylindrical containers at $200-250^{\circ}\text{C}$. The carbon monoxide returns into the circuit to form further iron carbonyl.

Flow diagrams of the high pressure apparatus, the pressure reducing plant, are available at the B.O.T. See p.33.

Raw Material: The raw material mostly used at I.G. Oppau for carbonyl production was iron scrap containing 1% of added sulphur as FeS_2 . This was produced at Duisberg by melting at $1200-1300^\circ\text{C}$ and shottling in water.

When production at Duisberg was held up, I.G. Oppau produced 3% sulphur slabs about 2" thick, and broke these down to hazel nut size in jaw crushers. Efforts were made to use scrap iron free from chromium, molybdenum, etc. as these lowered the rate of iron carbonyl formation.

The Procedure for Iron Powder Production is as follows. The filtered carbonyl is fed into the vaporiser so that it meets a tangential stream of NH_3 at the point of entry into the decomposer. The decomposer temperature at top, middle and bottom (exit) is 260°C , 280°C and 290°C respectively. The temperature of the heating gases in this same order is 440°C , 460°C and 360°C (exit).

The high pressure part of the plant, in which the carbonyl is formed, is made up of high pressure furnaces, preheaters for heating up the circuit CO, a heat exchanger, a circuit pump, a cooler and two vessels for the collection of liquid carbonyl. CO is supplied to the high pressure plant in a compressed state by the CO compressors.

The low pressure plant, in which the thermal decomposition of the iron carbonyl takes place, consists of decomposers powder removers, filters to clear the CO from the gas borne powder and CO blowers.

The decomposers are operated to produce only one quality of powder, i.e. Grade E, from which all other powders are produced by cyclone classifiers and/or decarburisation. The powder from the decomposer is sieved through a rotary sieve having 4900 holes per sq. cm.

Ball Milling. Certain of the powder grades are obtained by ball milling the standard Grade E powder which emerges from the decomposer. Milled E powder is termed EN, the suffix N or NN indicating ball milling.

For the milling operation, 1100 Kg of iron powder is ground by 1100 Kg of triple sized balls. Milling is, for safety reasons, carried out in a nitrogen atmosphere. Although I.G. Oppau had not suffered any explosions during grinding in air, Dr. Schlecht would not risk this process. The sale of unmilled powder has been discouraged during and since the war. The prices of these were frozen in 1939 and it has proved unprofitable to market them.

Decarburisation: Powder Grades C Normal and C Stabil are produced by the hydrogen decarburisation of Grade E powder, Grade C Stabil also receiving a pre-heating in nitrogen for two hours at 490°C.

The furnaces used for decarburisation or preheating in nitrogen are resistance heated, the furnace being divided along its length to permit its being pulled away from the decarburisation chamber in order to facilitate cooling. The decarburisation chamber is 10' long x 4' wide x 6" deep.

The charge is spread on trays with rollers to permit free movement. The furnace is held at 430°C, the temperature being measured by means of six thermo-couples evenly spaced along the furnace. Hydrogen is passed for the requisite period, then the heat is cut off and the furnace bodies are pulled away from the chamber.

The powder as removed from the decarburisation chamber is lightly sintered, but it is readily disintegrated in jaw crushers. It is then ball milled for 20 hours (1100 Kg. powder and 1100 Kg. balls) in a nitrogen atmosphere, and then passed through the rotary screen. The finished powder should contain between 0.02% and 0.04% carbon.

Preheating in Nitrogen: For C Stabil powder the procedure is generally similar to the process aforementioned, except that nitrogen is passed over the powder at 490°C, followed by hydrogen. The time of decarburisation is thirty hours and thereafter the treatment is as stated above for C Normal powder, namely, cooling, jaw crushing, ball milling, screening and mixing.

Cyclone Classification: This was carried out mainly in a double separator. The powder in a nitrogen atmosphere is fed into the top of the first chamber where it falls through the blades of a fan which acts to agitate the powder and distribute it well in the gas. A relatively slow gas current is maintained and the heaviest particles fall direct to the bottom of the first chamber, whilst the lighter part is bled off with the gas into a cyclone separator. The material separating from the cyclone is the normal production.

Grades of Carbonyl Iron Powder

A list of the powders produced, together with their electro-magnetic characteristics as determined under standard I.G. test procedures (see later), are given in Table 2. Many of these powders were produced only in very small quantities and the production of certain of them was discontinued. The main powders produced were Grades E, EN, C Normal, C. Stabil, and H. Small quantities of Grade Ferr. Red. B. and C. were produced and sold for pharmaceutical purposes.

Approximately 80% of total production was in Grades C Normal and E (or EN), these being produced in equal proportions. C Stabil was the powder next in importance regarding quantities, and then H. Grade EN powder was identical to Grade E, except that it had been ball milled, the milling being denoted by adding N (or NN) to the grade letter.

Grade A Powder

By means of the normal carbonyl process a crude iron powder called Grade A was produced. There was in Germany no large scale application for the powder in this state although a few experimental lots were supplied for catalytic purposes.

Grade C Powder.

The principle users of this grade were Siemens and Halske and A.B.G., who used it for low frequency cores for telephone work. This applied both to the development and maintenance of normal telephone circuits in Germany and to low frequency service requirements. C Stabil was stated to have the same applications as the C Normal powder, but owing to its higher magnetic stability it had certain advantages in the neighbourhood of electrified cables.

After 72 hours the C Normal powder lost 2% of its permeability. C Stabil lost 0.5% after 5 minutes but then sustained no further loss.

Grade E Powder.

The use of this powder was well established before the war for high frequency applications, particularly in conjunction with radio work. Large quantities of the E Grade powder were consumed in radio equipment for the Luftwaffe, and it is understood that it made the bulk of the higher frequency (radio frequency) applications. It has already been explained that Grade EN powder is normal E powder ball milled.

This powder is milled in cement type ball mills using balls of three sizes 75", 50 & 25 m.m. dia.) for 24-48 hours at 5-10 turns per minute. The charge consists of 1100 kgs of Powder and 1100 Kgs. of balls. The mills are lined with tungsten manganese steel and milling is carried out in a nitrogen atmosphere. This process breaks down the aggregates. The EN Powder contains more fines than the Grade E powder and has a maximum particle size of 8μ . It is not necessary to anneal this powder before use.

Grade H Powder

A very limited quantity of this grade was used in Germany as in this country, for highest frequency applications,

particularly in conjunction with Radar. Details of this grade are given in the table, and it represented the highest quality powder produced. It was obtained from that portion of the E powder which was caught in the Beth filter.

Grade P. Powder:

This powder which was used for Papin loading of submarine cables was made from Grade A Powder in the same manner as C Normal powder was made from Grade E Powder. If it was desired to reduce the oxygen content of this powder the treatment in wet hydrogen was followed by a similar treatment with dry gas (about 10 hours in each).

Grade S Powder:

This powder consisted of the larger fractions of Grade E, i.e. the residues after Grade H. powder has been removed. It was used for loading coils for local circuits and for radio direction finding loops.

Grade MR Powder:

Only very small quantities of this powder were produced, its properties being equal to Grade A. It was stated to be particularly suitable for electro-magnetic testing for cracks in steel and welded joints.

Grade Ferr. Red. Powder:

These powders are only those which fail to comply with the electro-magnetic specifications, and as shown in the table, were used for the preparation of pure salts and catalysts, and for pharmaceutical purposes.

Electro-Magnetic Testing of Carbonyl Iron Powders

It is evident that routine tests carried out at Oppau are very comprehensive and that a considerable amount of equipment is involved. Detailed information of the testing procedure, including preparation of powders and cores, is contained in a report prepared by Dr. Schlecht and his colleagues.

The position may be summarised as follows:-

The quantities measured are:-

- (a) Permeability
- (b) Eddy-current loss factor
- (c) Hysteresis " "
- (d) Residual " "
- (e) Stability " "
- (f) Damping (the inverse of 'Q')

The main equipment consists of:-

- (1) Insulation mixing machine
- (2) Press (300 tons) for toroidal and 'E' test cores
- (3) Toroidal winding machine
- (4) Audio frequency measuring bridge with oscillator, amplifier, filters, etc., for (a), (b), (c) and (d)
- (5) Special bridge for hysteresis of H powder
- (6) Magnetising equipment for (e)
- (7) Q meter (0.1 to 10 MC) for (f)

This equipment takes up a considerable amount of space and a qualified electrical engineer is necessary to supervise testing and maintenance.

A certain amount of variation in quality is met with in production; to overcome this, stocks were held consisting of individual batches with measured properties. When an order was received requirements were usually specified in detail and a suitable batch chosen, or sometimes a mixture of batches was made. Test results were submitted to the purchaser and also a sample; the latter was tested by the purchaser and if results agreed and were satisfactory, delivery of the batch was proceeded with.

It should be borne in mind that the most exacting requirements were for grades for loading coils and carrier frequency requirements, e.g. C Normal, C Stabil and H. These uses accounted for roughly 50% of the total output, the main consumer being Siemens and Halske.

For radio manufacturers it was only considered necessary to comply with a specification for overall loss measurement (damping or alternatively 'Q' factor) and effective permeability. The larger 'E' type test core as used by I.G., however, is more sensitive to quality variations. Detailed routine tests were made on all powders, but these were not, in general, submitted to radio manufacturers.

Details of the various core grades, together with their permeabilities, insulation, percentages, pressures and loss specifications, are given below:-

TABLE 3

<u>Powder</u>	<u>Insulation</u> <u>%</u>	<u>Pressure</u> <u>Kg/mm²</u>	<u>μ</u>	<u>w</u>	<u>Hc</u>	<u>n</u>	<u>Stabil-</u> <u>ity</u>	<u>Damping</u> <u>%</u>
C Normal	0.8	15000	53	0.7	55	6	2%	-
C Stabil	0.8	15000	48	0.7	42	6	1%	-
E	4	15000	16	0.7	5	1.5		
		3000						0.33
EN	4	15000	15	0.7	4	1.5		
		3000						0.30
H	4	15000	14	0.7	2	1		
		3000						0.28

μ = Permeability;

w = Eddy-current loss;

Hc = Coercivity

n = Initial factor;

Stability is a special test applied to loading coils. Percentage damping corresponds really to the inverse of (Q) factor.

With regard to the values given for w, it is suspected that these are not true values and that large corrections for winding loss should be added; this might account for only a single very high value being given for each powder, and, in actual fact, it would be expected that the values of w vary considerably for the different grades.

Carbonyl Iron Powder - German Prices

The prices of the various grades of iron powder produced at Oppau were frozen by order of the German State in 1939, and present production is being sold at these same prices by order of the French control authorities. Details of the prices of the various grades are shown in Table 3, but it should be noted that only the following are at present in production and represent the more important commercial grades: AS, EN, CNN, CN Stabil, CNS and H.

TABLE 3

German Prices, Carbonyl Iron Powders

AS	4 R.M./Kg.
EN	4 "
CNN	5 "
CN Stabil	5 "
CNS	4.5 "
H	4.5 "

Oppau Iron Powder Sales

No specific information was obtained on this point. The I.G. Farben Sales Organisation (M Division) was normally located in Berlin and was subsequently moved to Kothlen-Anhalt, near Magdeburg, and the main records were kept there.

Applications of Carbonyl Iron Powder

No applications for carbonyl iron powder were introduced in Germany during the war, other than for electrical purposes, and nothing very novel was learnt with regard to electrical applications.

Grade E or EN powder was mainly used for high frequency purposes, radio receivers and transmitters, low hysteresis induction coils, loading coils, etc. Grade C powder was used for audio frequency loading coils, filter coils, choke coils, etc. Grade H was used for purposes similar to those of E or EN, but was especially suitable for carrier wave frequencies and

and more particularly for uses over 100 MC/second.

Dr. Schlecht admitted that several nickel-iron materials would have been more economic to use for certain telephone and radio purposes than carbonyl iron powder, but the use of the latter was enforced under the stress of Nazi war economy owing to the nickel conservancy policy. He felt very strongly that there is a field for carbonyl iron, although this may be more limited than that available in wartime Germany and that it has definite advantages in special applications such as loading coils for multi band telephone cables.

A further field of commercial application is in the manufacture of carbide tipping tools where carbonyl iron has been found entirely suitable in conjunction with titanium carbide. At the present time approximately 4 tons per month is being delivered to the British Control Zone of Germany, especially Krupp, Widia Department.

Although the universal use of lead tetra-ethyl checked the use of $\text{Fe}(\text{CO})_5$ as an anti-knock for petrol, Dr. Schlecht believes that it is likely to return to favour in view of its less harmful effects in city atmospheres. He stated that the main disadvantage found at the time was the deposition of red powder on the sparking plugs, but he has heard that $\text{Fe}(\text{CO})_5$ is now being introduced into the U.S.A. and believes that some method for avoiding this deposition must have been evolved meantime.

MISCELLANEOUS

COBALT

Cobalt production in the I.G. organisation was at Bielefeld, the raw material being an impure cobalt oxide obtained from Duisberg Kuppenhutte, in which I.G. held about 90% interest.

The impure cobalt oxide was produced at Duisberg from copper containing roasted pyrites which were received from sulphuric acid plants where Rio Tinto pyrites had been treated. The copper production amounted to about 10,000 tons per annum, and in good years some 80/100 tons of cobalt (in oxide) was extracted.

Briefly, the treatment of the roasted pyrites comprised:-

- (1) Roast with potassium chloride
- (2) Leach with dilute sulphuric acid
- (3) Precipitate copper from the mixed copper chloride and sulphate solution with iron scrap.
- (4) Precipitate from the solution from (3), by means of lime, a cobalt slime containing also copper, manganese and iron
- (5) The cobalt containing slime was then treated chemically to separate the various metals, and the cobalt was recovered as an impure oxide which was sent to Bielefeld.

Before Bielefeld undertook the production of pure cobalt powder, Duisberg supplied crude cobalt concentrates to the market, but this practice was abandoned when Bielefeld started making, since a much better profit could be obtained with the pure metal.

At Bielefeld the oxide was purified by chemical means, the impurities being precipitated out. The pure oxide thus produced was reduced in hydrogen to give pure cobalt metal.

No cobalt salts were made at Bielefeld. The pure metal was sold, almost entirely, for carbide tool tip manufacture, Krupp being the largest buyer for their Widia production. A small quantity also went to Deutsche Edelstahlwerke for the same purpose.

Originally Bielefeld made molybdenum and tungsten powders, tungsten carbide, titanium carbide, etc., and it was because of their activities in these fields that they found the need for a pure cobalt metal. This led to their entering the business.

Cobalt Production

An I.G. plant at Griesheim (near Frankfurt), which had been much longer established than that at Bielefeld, produced the other cobalt products in which I.G. were interested. The raw material comprised miscellaneous parcels which came on the market chiefly from Katanga (Hoboken), etc. The main products were cobalt acetate and cobalt formate used for paint driers and in linoleum manufacture.

Dr. Schlecht said that no other products were manufactured and pointed out that cobalt containing raw materials were always in very short supply in Germany. When the Petsamo mines became available, Dr. Schlecht started some experimental work with a view to extracting the cobalt. He estimated that some 200 tons cobalt could be obtained along with the 10,000 tons nickel expected per annum from the Petsamo ore.

Dr. Schlecht was not aware of any I.G. production of cobalt oxide or hydrate for the market. Cobalt oxide might have been supplied in small quantities from Griesheim. If the hydrate was made by I.G. it could only have been in limited amounts according to Schlecht because he had never heard this product discussed at the half yearly I.G. "metal interests" meeting.

Soligen Driers

Dr. Schlecht said that Soligen Driers were made at Griesheim by the oxidation of paraffin either by (a) nitric acid or (b) air or oxygen. At the present time one or both of these methods are being employed in plant at Oppau. This plant is in close proximity to the iron carbonyl plants for which Schlecht himself is responsible, but he had no detailed information on the process.

He believed that oxidation with oxygen or with air enriched with oxygen is the probable method used, because there is a surplus of oxygen from the fertiliser plants. The plant had been destroyed by bombing, but was now repaired and in operation. The product is used mainly for soap manufacture, although he believes attempts have been made to use it for producing edible fats. In connection with the latter, Dr. Schlecht was unable to say what catalysts had been employed.

NICKEL IRON POWDERS

Because of the practical ease with which nickel and iron powders can be mixed and thermally diffused into alloys, Dr. Schlecht had given little attention to the production of nickel-iron powders from mixed carbonyls. Small quantities of 50/50 and 78/22 nickel-iron had, however, been made. The mixed carbonyls were fed into the vaporiser which was kept dry to prevent selective evaporation, and the decomposer was operated in the normal manner. The resultant powder was not alloyed, but could be alloyed by further treatment. Dr. Schlecht had very little knowledge of the electro-magnetic properties of these nickel-iron powders, the use of nickel being prohibited in Germany for these purposes and little development work was accordingly carried out.

COBALT CARBONYL

Only experimental quantities of this had been made, although some had been recovered from the distillation of crude nickel carbonyl. It was obtained by the well known method, i.e. treatment of reduced oxide with CO at 150-200 atms. pressure and at 140°C.

Cobalt-nitroso carbonyl had been made experimentally by Dr. Schlecht at 10 atms. pressure by passing 90% CO, 10% NO mixture over reduced cobalt oxide. The compressed gas mixture, however, exploded spontaneously, and the experiment was abandoned. A.E. Wallis believes that possibly cobalt might be extracted economically as nitroso-carbonyl from cobalt residues, etc.

MOLYBDENUM CARBONYL.

This was produced only in laboratory quantities, i.e. one or two pounds, and by acting upon molybdenum-copper mixtures with CO at 200 atms. and 230°C. The reaction gases were cooled in liquid nitrogen to obtain a maximum yield of Mo(CO)_6 . The molybdenum-copper mixture was made from the chlorides by NH_3 precipitation, followed by H_2 reduction at 500-550°C. Dr. Schlecht expressed the view that this method of production is not economical compared with present normal methods.

TUNGSTEN CARBONYL.

A few grams of this were made under the same conditions as Mo(CO)_6 .

PATENTS

Dr. Schlecht had not brought any copies of the various recent I.G. Farben patents with him on his visit to the U.K., but he referred briefly to the following subjects which he recalled as having been covered by patents.

A process of producing nickel carbonyl from complex nickel compounds by injecting chlorine through a solution of nickel salts in water. (b) A treatment of nickel copper sulphide matte by which it is roasted to remove sulphur and subsequently reduced. (c) Granulating nickel matte. (d) Desulphurisation of nickel matte by blowing air into the converter followed by reduction.

The dilution of carbonyl vapours by means of CO before entering the decomposer or by reducing the pressure in the decomposer below atmospheric pressure. (f) A process for the manufacture of sintered materials which are permeable to liquids with a specific application to fountain pen nibs. (g) A process for producing aluminium nickel iron for example, by first making an alloy of aluminium and nickel and subsequently sintering this with nickel and iron powders. (h) Desulphurisation of nickel powder by treating in hydrogen and steam for a fairly long period of time with a view to improving workability. (i) Removal of carbon from iron powder by adding correct amount of iron oxide and heating. (j) A process for the manufacture of magnetic cores from metal powders.

The diagrams to which reference is made on p.20 have been lodged with:-

The Board of Trade,
German Division (Documents Unit),
Lansdowne House,
Berkeley Square, W.1.
Telephone: Grosvenor 4060, Ext.2923.

For permission to inspect these documents please quote
BICS/DOCS/NIL/3017.

Powder	Particle Size	Method of Preparation	% C	% N ₂	u	w	h	n	Stability	% Damping $\alpha = 235 \text{ m.}$	Special Uses
A	10 μ	Decomposition of Fe(CO) ₅ without ammonia.	1	-							Main material for sintered products and for the preparation of powder P, and also of Ferr. Red.
E	8 μ	With addition of NH ₃	0.7	0.6	16/18	0.5	4/5	1		0.33	For high frequency purposes, radio receivers and transmitters, low hysteresis induction coils, loading coils, etc.
EN	8 μ	From E powder by ball milling	0.7	0.6	15/17	0.5	3/4	1		0.30	The same uses as E. Better Q value.
H	4 μ	That portion of E caught in the Beth filter.	0.6	0.5	14/15	0.5	1/2	0.7	After 5' 0.5%	0.28	The same as E and EN but better hysteresis and Q properties. Especially suitable for carrier-wave frequencies and more particularly for uses over 100 mc/second.
C Normal	8 μ	From E powder by heating in H ₂ to 430° C.	<0.1	0.02	53	0.6	55	5.5	After 7' <2%		For audio frequency, filter coils and choke coils (Drosselspulen).

Table 2.

Powder	Particle Size	Method of Preparation	% C	% N ₂	u	w	h	n	Stability	* Damping $\gamma = 235 \text{ m.}$	Special Uses
C Stabil	8 μ	From E powder by first heating in N ₂ to 490°C, then in H ₂ to 390°C.	(0.1	0.02	48	0.6	42	5	After 5' (1%		The same as C Normal but of higher magnetic stability, i.e. near to (alongside) electrified cables.
S	8 μ	Larger particles by cyclonic separation from E powder. Heated in H ₂ to 490°C.	(0.1	0.01	60/65	0.6	80/120	6			The same as C Normal but especially where higher permeability is demanded.
P	10 μ	From Powder A, by heating in H ₂ to 430°C.	(0.1	-	25	0.6	7	6.5			For loading coils for multi-band telephone cables (Mehrband Teleforkabel).
Ferr. Red. A	10 μ	Prepared as Powder A	1								For the preparation of pure salts and catalysts.
Ferr. Red. B	10 μ	Prepared as C Normal	(0.1	0.02 (0.15% Ni)							For pharmaceutical purposes (ferrous iron medicines).
Ferr. Red. C	10 μ	Prepared as C Normal of selected nickel content.	(0.1	0.02 (0.05% Ni)							As Ferr. Red. B.

Table 2 (contd.) * "Damping" Reciprocal of Q