B.I.O.S.—FINAL REPORT No. 468

ITEM Nos. 21, 22

THE MANUFACTURE OF SYNTHETIC CRYSTALS IN THE PLANT OF I.G. FARBENINDUSTRIE OPPAU — LUDWIGSHAFEN

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

The Manufacture of Synthetic Crystals in the plant of I.G. Farbenindustrie Oppau - Ludwigshafen

Reported by
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BIOS Target Nos.
22/1(f), 21/22(a).

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BIOS FINAL REPORT NO. 468

entitled

"THE MANUFACTURE OF SYNTHETIC CRYSTALS IN THE PLANT OF I.G. FARBENINDUSTRIE OPPAU - LUDWIGSHAFEN."

E R R A T U M

It is brought to notice that

(a) Fig. 1. shown as 1/5 Full Scale - should read 1/6 Full Scale.

(b) Figs. 2 and 3 shown as Full Scale should read 4/5 Full Scale.

Insert in: QC391/p 255.

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Illustrations

1. Introduction

In November 1945, Dr. Karl H Luft was brought to England for interrogation on his infra-red gas analyser and a recording infra-red spectrometer with which he had been working. In the course of interrogation he disclosed that I.G Farbenindustrie Oppau-Ludwigshafen had been successful in making very large synthetic rock salt crystals. Since the writer had already planned to go to Germany on another mission in the locality of Ludwigshafen, it was decided that he should investigate the process on the spot.

1.1 Personalities

The equipment and process originally described by Kuropulous have been further developed and operated by Herr Ingenieur Korber whose private address is Plankstadt, Karl Theodorstr.7. (bei Klee) Heidelberg - Schwetzingen. The work started under Gmelin's direction about 17 years ago and continued until interrupted by bombing in the Laboratory of the Betriebskontrolle of I.G Farben Oppau. Dr. Ernst assisted by Dr. Luft supervised the work latterly.

2.1 Description of the Equipment

In December 1945, the equipment was located on the ground floor of the bombed out Betriebskontrolle Laboratory which is on the left hand side of the road about 300 yards inside the Oppau plant from the Ammoniakstr. gate. The equipment consists of seed crystal gantry, two melting furnaces, three or four tempering furnaces and associated electrical equipment etc. Fig. 1 shows the general arrangement of seed crystal gantry (A) and melting furnace (B). Molten purified Sodium Chloride is maintained at a temperature of 860° to 900°C in a quartz crucible (C) of wall thickness about 1.5 cm which is contained in the melting furnace.

The furnace, which is lagged with asbestos, has four separate electric heating zones (D_1 D_2 D_3 and D_4) and the heating currents to these four separate elements are independently controlled thermostatically by mercury relays operated by the four platinum resistance thermometers (E_1 E_2 E_3 and E_4). The construction of these thermometers is illustrated in Fig. 3. They are wound with 0.01 mm platinum wire on an asbestos former to a cold resistance of 100 ohms and protected against damage by a quartz sheath. The thermometers are necessarily located on the outside walls of the crucible to avoid temperature and convection disturbances to the melt. The top of the crucible is closed with two semi-annular quartz discs (F) about 1.5 cm thick and great care in the preliminary adjustment of the heating currents is necessary to compensate for heat losses from the top of the crucible and ensure that there are no convection currents in the melt due to temperature gradients in a vertical direction. The seed crystal (G) is turned to the shape and held in a chuck (H) as illustrated in Fig. 2. The top of the seed is separated from the watercooled base of the chuck by a steel cylinder (I) and cooling water is introduced through the pipe (J). The chuck and its support are rotated one revolution per two hours by the synchronous motor and gearing (K) and the cooling water escapes by the pipe (L).

The rotating chuck holder with its drive is supported by the gantry (M). The gantry beam can be raised or lowered by the hand wheel (N) and each pillar can also be raised 1.0 mm per hour by worm gearing driven by the synchronous motors (O_1 and O_2).

The tempering furnaces are similar in outward appearance to the melting furnaces. However, there is only one heating element which embraces all the sides and bottom and the top is closed with a heavy heat insulating lid. The synchronous motors, relays and thermometers were supplied by Hereaus, Frankfurt-Hanau.

2.2 Description of the Process

With water cooling and motors O_1 and O_2 turned off, the melt in the melting furnace is brought to a temperature of about 870°C. With the motor K switched on, the seed crystal is then slowly (over ½ hour) introduced into the air space between the melt and the quartz cover where it is allowed to remain for two or three hours to attain temperature equilibrium.

The seed crystal is then slowly moved downwards until it is in contact with the surface of the melt. If the temperature is too low, a crop of small crystals will form on the point of the seed while if it is too high, the point of the seed will melt away rapidly. The correct temperature is that at which the changes 1 to 3 in Fig. 4 occupy about 2 hours. Inspection is by moving one of the quartz covers and the attainment of the correct temperature is largely a matter of experience. If the temperature is incorrect, the heating current is adjusted and the seed crystal lowered further either to melt the small crystals formed or to present a fresh seed surface to the melt.

When the correct temperature is reached, the cooling water is turned on very slowly; at first, each drop is turned to steam as soon as it reaches the chuck base and the crystal starts growing as illustrated in Fig. 4 stages 4 and 5. The flow of cooling water is gradually increased but cautiously to prevent the formation of small crystals. If such should happen, the flow is reduced and they are melted off. When the mushroom shaped crystal as in stage 5 has grown to the required diameter, the lifting motors (O_1 and O_2) are switched on and the crystal starts growing vertically as illustrated in stage 6. The flow of cooling water must be gradually increased during the period of vertical growth while observing the precautions against overcooling. The height of the cylindrical crystal is measured by a platinum dip wire a fixed double bend of which can be located on the top surface of the cylinder.

When the required height has been obtained, the cooling water is turned off and the crystal removed from the melting furnace by the hand wheel (N). The melting furnace is moved aside and the tempering furnace preheated to 600°C and containing a conical pile of purified sea sand (quartz) covering the bottom is substituted. The crystal is immediately lowered into the tempering furnace to within 2 or 3 cm of the tip of the pile of sand. The neck (i.e the cylindrical part of the old seed crystal is then cut with a sharp chisel and the crystal falls gently onto the pile of sand.

At the moment it leaves the melting furnace, there is a temperature gradient from about 400 to 680°C and the bottom of the crystal is quite plastic: The use of purified quartz as a bed for the crystal largely prevents the incorporation of impurities which might cause differential cooling contraction. The temperature of the crystal is measured with at Platinum - Platinum-rhodium thermocouple lying on the upper surface, thermostatically controlled and recorded and a second thermocouple measures the furnace temperature.

The oven is maintained at 600°C for 8 or 10 days to get rid of any thermal strains in the crystal after which slow cooling may be started. The actual rate of cooling is dependent on the size of the crystal and should be varied from 2°C/hours for a crystal of less than 10 cm diameter to ½°C/hour for one of from 15 to 30 cm diameter. Crystal and furnace temperatures must be the same throughout the cooling and the furnace should be of such a size that it is approximately filled by the crystal. After reaching room temperature, the crystal should be allowed to remain in the oven for 3 or 4 days before removal.

A variety of crystals have been made with the equipment as shown in the following table and it was pointed out that the sizes given only indicate the maximum size actually grown and do not represent the limit of the capabilities of the equipment.

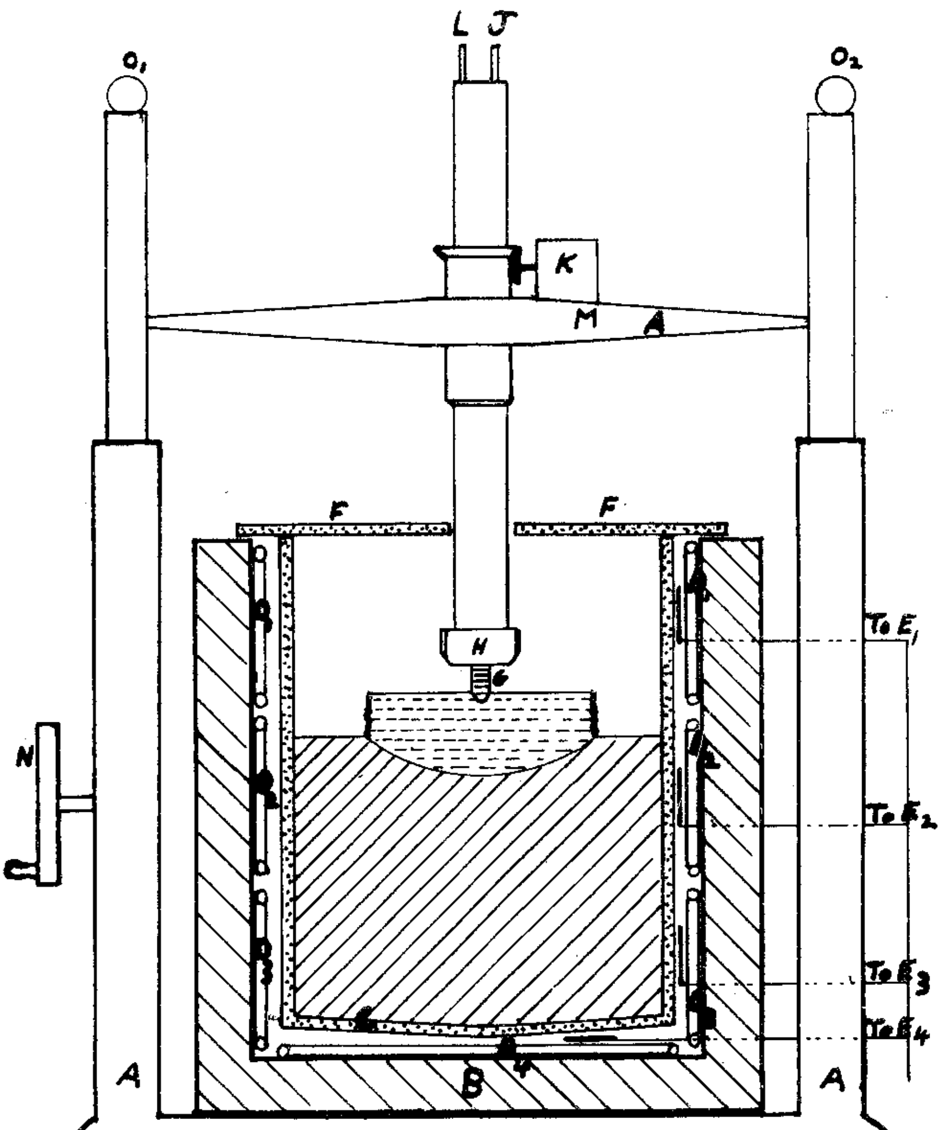
Table

Substance	Diameter of Crystal
Sodium Chloride	30 cm
Potassium Chloride	25 cm
Sodium Bromide)	9 cm
Potassium Bromide)	possibly more
Calcium Fluoride	tried
Lithium Fluoride	4 cm (about the limit)
Potassium Iodide	3 - 4 cm
Silver Chloride	3 - 4 cm
Potassium Chloride + 10% Silver Chloride or Lead Chloride	samples

Substance	Diameter of Crystal
Lithium Fluoride) +2% Magnesium Fluoride)	Samples

In the case of fluorine compounds, all parts which come into contact with the salt or its vapour must be made of platinum.

FIG. 1.



MELTING FURNACE AND CRYSTAL GANTRY
$\frac{1}{5}$ FULL SCALE (APPROX.)

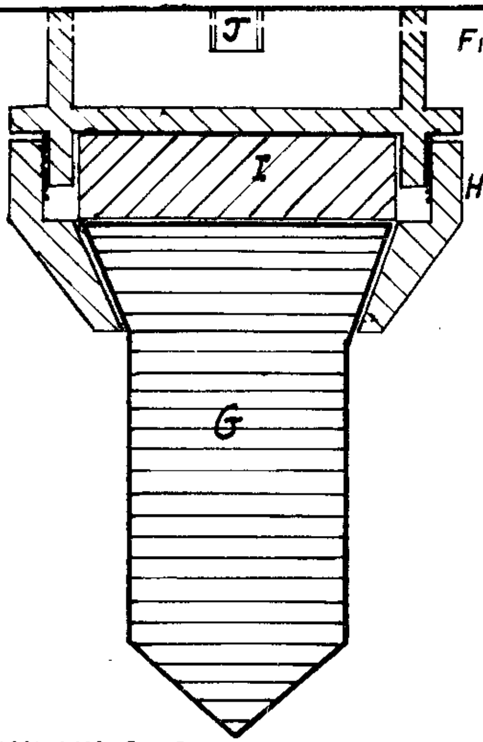
	Datum	Name
Gezeichnet		
Geprüft		
Normgepr.		

Maßstab

Ersatz für:

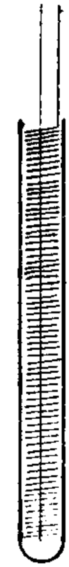
Ersetzt durch:

FIG. 2.



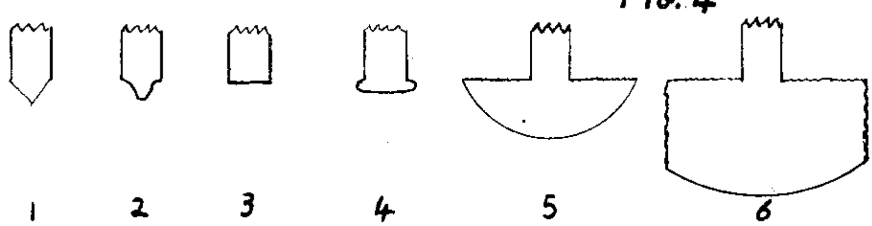
CHUCK AND SEED CRYSTAL
FULL SCALE

FIG. 3



PLATINUM
RESISTANCE
THERMOMETER
FULL SCALE

FIG. 4



COURSE OF CRYSTAL GROWTH

	Datum	Name		
Gezeichnet				
Geprüft				
Normgepr.				

Maßstab		
		Ersatz für:
		Ersetzt durch:

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