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NEW LIQUID INCENDIARY AGENT

(ClF_3), 23 April 1945

War Dept, Combined Intelligence Objectives Subcomm. Report #36

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

7 May 1945

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INFORMATION ON A NEW LIQUID INCENDIARY AGENT (ClF_3).

23 April, 1945.

Reported by

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7 May, 1945.

CIOS Black List Item - 17
Flame and Incendiaries

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE
G-2 DIVISION, SHAEF (REAR) APO 413

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INFORMATION ON A NEW LIQUID INCENDIARY AGENT (ClF_3)

1. INTRODUCTION.

a. The information contained in this report was obtained from Dr. L. Klebert, Director of Inorganic Department of the I.G. Farbenindustrie plant at Leverkusen, Germany and Dr. Erich Noack, who is a member of Dr. Klebert's department, during conversations on 23 and 25 April, 1945.

b. Prior to 1937 a Dr. Kwasnik began an intensive study of fluorine compounds since the I.G. plant at Leverkusen was interested in obtaining a good fluorinating agent for the fluorination of hydrocarbons. Investigations were made of halogen trifluoride compounds which had been described previously in the literature. (Ruff and Krug; Über ein neues chlorfluorid - ClF_3, zt. Anorg. Chem.1930, 190, 270; Ruff; Neues aus der Chemie des Fluors, zt.f. Angew. Chem. 1933, 46, 739.) Iodine trifluoride was prepared and tested for the fluorination of hydrocarbons with some success. As a matter of scientific interest, chlorine trifluoride was prepared and found unsatisfactory for the purpose just mentioned. The Inorganic Division had found no use for ClF_3 and had no interest in it until 1937 when the German Army Ordnance inspected the laboratory preparing the material. The Army subsequently asked for samples at intervals; the first sample was sent about four years ago, the last in 1944. During this period the laboratory prepared 3 to 5 tons of ClF_3. It is believed that the German Army intended to use ClF_3 in shells against aircraft and tanks. The unique properties of the substance would be peculiarly suited against such machines since organic matter would be immediately ignited and glass or plastic windows would be permanently etched and rendered opaque.

2. PROPERTIES OF ClF_3.

a. The structure of the substance is not known. It is a liquid with a boiling point of 12° C. When stored in iron pressure cylinders, the material is stable and can be handled and shipped without difficulty.

b. The substance combines with all organic and a number of inorganic compounds so vigorously that the heat generated will generally cause the material to burst into flame. Combustible organic compounds will then continue to burn in the normal manner. Non-combustible inorganic substances cease to flame as soon

as the ClF_3 is dissipated. Glass, for example, would be badly etched and rendered opaque by contact with ClF_3. Glass wool is said to burn with a flame in the presence of ClF_3 and the reaction with water is so vigorous as to give off incandescent gases. The reaction of ClF_3 with glass and water is illustrated as follows:-

$$SiO_2 + ClF_3 \rightarrow SiF_4 + O_2 + Cl_2 + \text{heat}$$

$$H_2O + ClF_3 \rightarrow HF + O + Cl_2 + \text{heat}$$

3. PREPARATION OF ClF_3 AT LEVERKUSEN.

a. All the necessary apparatus for manufacturing on a laboratory scale was contained in a room with floor dimensions of about 8 x 16 ft. This small plant could make 3½ pounds of ClF_3 per hour. The process of manufacturing is as follows:-

b. Fluorine is produced by electrolysis in a cell made of metallic magnesium. Carbon anodes are used with the metallic vessel serving as the cathode. The electrolyte, which is KHF_n) is kept at 100°C and is not dissipated since liquid HF is continuously fed into the cell and broken down into H_2 and F_2. A sketch of the cell and detailed description of its operation have been obtained for comparison with those employed industrially elsewhere.

c. The gases from the fluorine cell are passed through a coil immersed in dry ice (-80°C) to remove HF. The fluorine gas is then mixed with chlorine gas in proper proportions to have an excess of fluorine at all times. A blue flame results at the point of mixture. The gases are then passed through a U shaped tube which is heated to 280°C. The ClF_3 thus obtained is condensed in a coil immersed in a -80°C bath and collected in iron cylinders. The cylinders are vented several times to allow HF, Cl_2 and F_2 to escape. The liquid ClF_3 is stable in such iron cylinders for long periods of time.

4. INDUSTRIAL DEVELOPMENT OF ClF_3.

a. No industrial development of ClF_3 was undertaken at Leverkusen. All information on this substance was given to the Laboratorium des Heereswaffen Amtes at Spandau, a suburb of Berlin; this is a laboratory of the Ordnance Department of the German Army. The Army name for the incendiary substance was "C-3" and the plant where it was subsequently manufactured was known as the "Seewerk".

b. The Army built a small pilot plant at Kummersdorf which is an artillery proving ground about 50 kilometers south of Berlin, slightly west of Route 96 near a town named Sperenberg. This plant was of about the same size as the one at

Leverkusen, and differed from it in no essential respect except that the fluoride cell was operated at a somewhat higher temperature. While it is not known when this plant was built, it was in operation in late 1943 when visited for a few hours by Dr. Erick Noack and Dr. Kwasnik, both of I.G. Leverkusen. Dr. Kwasnik has recently moved to the I.G. plant at Bitterfeld; he is a specialist on fluorides and did most of the development work on ClF_3 at Leverkusen. During its existence the pilot plant made several tons of ClF_3.

c. A production plant for ClF_3 of unknown capacity was built at Gottow which is part of the Kummersdorf proving ground. Drs. Klebert and Noack do not know whether this plant was ever completed or, if completed, whether any ClF_3 was produced. They feel sure that ClF_3 was never used by the German Army.

d. Dr. Klebert was questioned regarding the availability of samples of ClF_3. He said none was available and that the laboratory plant for ClF_3 at Leverkusen had not operated for almost a year. The plant could be reconditioned for the production of a few pounds of ClF_3 per hour within a month provided that Dr. Kwasnik could be brought back from Bitterfeld to do the work.

5. EFFECT OF ClF_3 ON HUMAN BEINGS.

a. No fatal accidents occurred during the work on ClF_3 at Leverkusen. However, experience was obtained with effect of the material on the skin. Contact was immediately followed by a flash flame which did not in itself cause an appreciable burn. However, the resultant formation of HF at the surface of the skin produced a chemical burn. These were treated by washing with buttermilk and bandaging.

6. RECOMMENDATIONS.

On the basis of the above statements, it is recommended that:-

a. Arrangements be made to produce samples of ClF_3 at the earliest possible moment to determine if properties are as described above.

b. Work on this substance be undertaken by C.W.S.

c. The ClF_3 plants at Kummersdorf and Gottow be considered

Black List targets and be investigated at the earliest opportunity.

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APPENDIX I.

TRANSLATION OF DESCRIPTION OF FLUORINE APPARATUS.

"FLUORINE APPARATUS NO. 3 - for 2000 Amp.

The cell vessel as well as the cover consists of Elektron. All the carbon anodes and the sheet nickel cathodes are connected in parallel and electrically insulated from the cell vessel and the cover respectively. To cool and homogenize the electrolyte (especially during the introduction of HF) (regeneration, see Appendix II), it is pumped through a lateral neutral compartment* containing a compressed air stirrer. A corresponding neutral compartment* on the other end of the cell contains a cooling coil (external cooling). The flowing electrolyte is led back from one end of the cell to the other through an Elektron tube beneath the cell. In this tube there is also a water cooled tube (internal cooling), The neutral compartment with the stirrer contains the HF inlet tube. The neutral compartment with the cooling coil contains a thermometer. In order to detect stoppages of the gas outlet tube, which may be caused by electrolyte dust, a slow stream of N_2 is led through the electrode compartment so that its flow may be observed in a bubble bottle. The usual diaphragms are omitted; the cell compartments are separated only in the portions wet by the melt. The electrolyte is a mixture of 1 pt. KF to 2.5 pt. HF. The working temperature is 80-100°C.

Because the man who did the experimental work is not available at present and because there are no written records, the above description may contain inaccuracies or small errors here and there.

* Presumably electrically neutral - i.e. containing no electrode".

APPENDIX II.

TRANSLATION OF OPERATING INSTRUCTIONS FOR FLUORINE.

"FLUORINE PREPARATION (OPERATING INSTRUCTIONS)

Very Confidential

1. Temperature control. Keep the electrolyte at 70-75°C.

2. Observe whether the stirrer is working satisfactorily.

3. Control the level of the melt at 4 cm below the cover.

4. Put the bubble bottle (a high pressure glass vessel filled with H_2SO_4) under nitrogen pressure. Notice whether there is any stoppage of the pipes.

5. Turn on the direct current machines in the order given by the numbers on the switches.

6. Turn on the current gradually. If the cell operation is not smooth load it with a maximum of 500 amps, and hunt for the source of trouble (level of electrolyte too low, stoppage of the pipes, partially opened valves).

7. When the cell is loaded with 500 to 800 amps, start the internal cooling (outlet temperature 34 to 38°C), with higher loading using external cooling also.

8. The cell is shut down in the reverse order: external cooling off, internal cooling off, current off, bubble bottles turned off, direct current machine shut down.

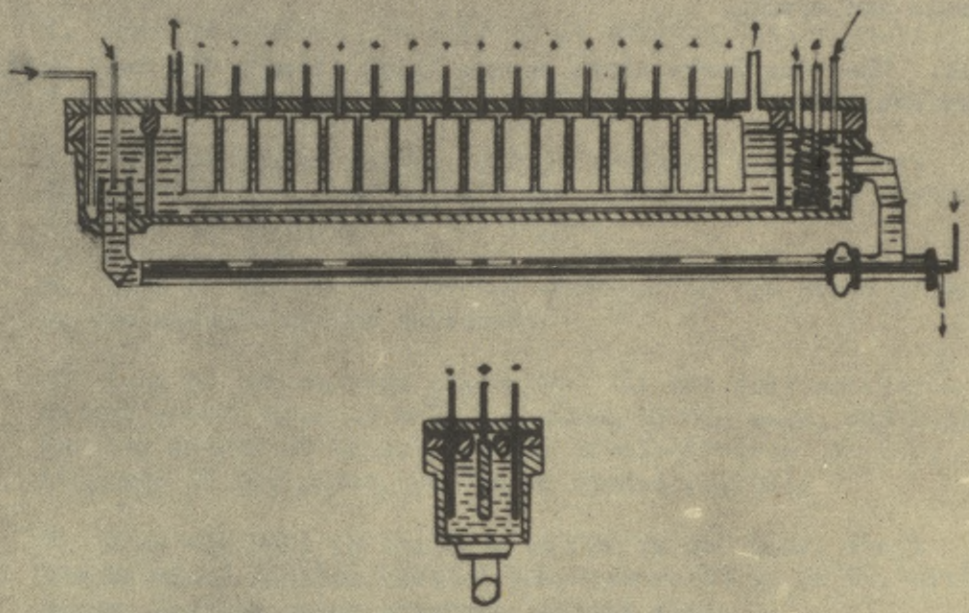
9. The regeneration of the melt is carried out as follows: The HF cans are heated to 40°C thereby the vapor pressure of HF becomes high enough to cause liquid HF to flow into the melt until the HF content of the melt has reached its original value. This can be done while the cell is in operation. A control analysis of the composition of the melt (Titration with N/1 NaOH and phenolphthalein) is necessary only at intervals of 6 months. (Composition KF 2.4, HF 2.6).

APPENDIX II. (Continued)

10. If, for any reason, the melt freezes in the circulating pipe (for example when the current fails), instead of cooling water steam is passed through the internal cooling coil until the electrolyte is again liquid and is pumped through the stirrer.

11. The plate built into the circulating pipe, (thermal expansion equalizing plate), must be continuously heated with gas as it will otherwise swell up with the freezing melt and crack.

12. When the Works alarm sounds (high siren), the cell must be shut down.*



PLUCKING CELL No. 3 2000-3000 AMP.

APPENDIX III.

TRANSLATION OF OPERATING INSTRUCTIONS FOR ClF_3.

"PREPARATION OF C.T.F.(ClF_3). (OPERATING INSTRUCTIONS).

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The cooling coil (for removal of HF from F), the condensing coil (for C.T.F.) and the storage flasks for C.T.F. were cooled with dry ice. The reaction oven is heated to 280°C and flushed with F. As soon as fluorine can be detected in the outlet tube, the flow of chlorine is started and regulated as follows:-

58	liters	Cl_2	per	hour	with	650	Amp	load	on	F cell
89	"	"	"	"	"	1000	"	"	"	" "
134	"	"	"	"	"	1500	"	"	"	" "
178	"	"	"	"	"	2000	"	"	"	" "

The maintenance of constant temperature in the reaction chamber follows automatically. While the cell is in operation, the liquid C.T.F. must be tested every 15 minutes by looking through the peep-hole to see that it is colorless and flowing off quietly. If the flow of C.T.F. is too slow, there is a deficiency of chlorine; if it is yellow instead of colorless there is either too much chlorine or a leak in the fluorine pipe.

As soon as one storage vessel is full, the valves on the condensing coil (for C.T.F.) are reset so that liquid C.T.F. flows into the second storage vessel.

The most important thing to do during the preparation of C.T.F. is to watch the apparatus to prevent stoppage of the pipes (formation of FeF_3). If a stoppage occurs, all the liquid C.T.F. that has collected in the cooling coil and pipes must be completely vaporized before repairs can be undertaken. If liquid C.T.F. leaks out, it is covered with a generous quantity of dry ice and the C.T.F. and CO_2 allowed to vaporize off in a hood.

As soon as a steel bottle is filled with C.T.F., it is closed with a degreased Hofer valve and allowed to come to room

temperature. A 10 atm pressure gauge is then attached to this valve and a second valve added after the gauge. The excess pressure is then carefully removed by venting (F_2,Cl_2,C.M.F.) until the pressure has dropped to 1.2 atm. The steel flasks are then ready for shipment.

Leverkusen, 26 September, 1944.

(signed) Kwasnik."