# Electrolytic Manganese & Manganese Dioxide from Low-grade Indian Ores

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The National Metallurgical Laboratory has developed a method of producing electrolytic manganese which can effectively deal with almost all kinds of low-grade manganese ores, securing 99.95% pure manganese metal. The authors discuss the method and contend that with low-grade ores having not more that  $30\%$  manganese, the cost of production of electrolytic manganese in India in a 10-ton-a-day plant is not likely to exceed Rs.  $1,000$  a ton. The work being done at the Laboratory to produce electrolyticbattery-active manganese dioxide from low-grade Indian ores, is discussed separately; it is stated that laboratory-scale experiments have successfully been completed and a semi-pilot plant is under development. The production cost of electrolytic manganese dioxide is reckoned at about Rs. 600 per ton, allowing margin for various factors; the price of the imported product is more than Rs. 1,000 per ton.

IN the past five years (1951 to 1956) a total amount of 6,06,4000 tons of manganese ores have been exported from India and foreign exchange equivalent to Rs. 853,200,000/- has been earned. Deposits of manganese ores are widely distributed in India. Important deposits are located in Madhya Pradesh, Orissa, Bombay, Mysore, Bihar and Andhra. About 85 to 95% of the ores mined are exported. Production at present is largely dependent on the demand in the foreign market; nevertheless, in recent years the production has shown a definite tendency to go up.



These production figures mainly refer to the metallurgical grade ores which have an export market. But during mining, almost an equivalent quantity of low-grade ores come up which are unsuitable for production of ferro-manganese. These in general contain less than 40% of manganese and are practically without any market. Table II shows the relative price and quality of the ores. It is obvious that ores containing 30% or less of manganese are at present without any value whatsoever.

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# TABLE II-QUALITY AND EXPORT PRICE OF MANGANESE ORES<sup>\$</sup>



Assuming that in mining for high-grade manganese ores the low-grade ores are produced in almost equal quantity the annual accumulation of the latter on the mine site is about 1.5 million tons per year involving about 0.4 million tons of manganese metal. If this amount of metal could be extracted as electrolytic manganese, it would be valued at nearly hundred and twenty crores of rupees according to present quotation of the commodity. This colossal wastage of national resources is eating into the vitals of the manganese mining industry every year. It should be well to remember that the known reserves of low-grade manganese ores in our country are estimated above 70-80 million tons.

At present about 85% of the high-grade metallurgical manganese ores are exported and the rest are mostly utilized by the iron and steel industries. Because of the high phosphorus content of Indian coals, the ferro-manganese produced in the country also contains high phosphorus and this is one of the reasons which stand in the way of production of standard-grade ferro-manganese. The high iron content of the manganese ores is another reason which makes ferro-manganese production difficult.

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As a result the development of the ferro-manganese industry in India has not been proportionate to the country's manganese and iron resources. Production and import figures in Table HI reflect the present state of the ferro-manganese industry.

TABLE III—PRODUCTION AND IMPORT OF FERRO-MANGANESE<sup>5</sup>

Year	Production	Import (ferro-
		alloys)
1952-53	*34,900	1.293
1953-54	$*1.221$	3.447
1954-55	43,513	3.567

#### \*Including spiegel

Of the two handicaps mentioned above, difficulties due to high phosphorus in coal may be minimized by using electrical energy for heating. In persuance of the targets for second Five Year Plan licenses have been issued for setting up of plants to nine parties for the production of 171,800 tons of ferro-manganese per annum. Actual production by 1960-61 is expected to reach 160,000 tons a year, of which 100,000 tons are meant for export. The major raw materials required for the same are given below.<sup>5</sup>

#### TABLE IV



Electrical power consumed per lb of ferromanganese calculated on the above estimate comes to 1.8 KWH. This point is important because electrical power is a compulsory substitute for coal. Further, only high-grade manganese ores can be used for this purpose and coke of satisfactory specification will have to be used. Producers of ferro-manganese will have to face these handicaps.

The Krupp-Renn and Basset process for production of ferro-manganese claims certain very attractive features, regarding quality of coal, ore and by-products. Since not much information is available on the process, the relative advantages cannot be evaluated. Claims put forward indicate possibilities of utilizing off-grade manganese ores and coking coal for the purpose of producing ferromanganese.

Ore-dressing methods are capable of upgrading certain kinds of low-grade ores and making them suitable for ferro-manganese production. But these methods have limitations and are not of much help in case the gangue materials are closely associated with manganese minerals. Thermal beneficiation with manganese minerals. method depending upon production of manganese rich slag is applicable to certain varieties of ores but the high phosphorus content of Indian coals is again a disquieting factor to be faced.

Some methods of upgrading manganese ores are based upon chemical treatment of the ore with

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sulphur dioxide, oxides of nitrogen, ammonium sulphate and the like whereby the manganese is dissolved out from the ore and precipitated out as oxides which are capable of being used in place of standard-grade manganese ores. In following these methods, a question naturally arises since manganese is to be used either as metallic manganese or as ferro-alloy in iron and steel industry—is it not advantageous to precipitate out metallic manganese instead of oxides from the chemical solutions obtained from ores'? This change in chemical dressing methods will do away with the disadvantages associated with reduction of manganese oxides to ferro-manganese. This is exactly where production of electrolytic manganese steps in with definitely advantageous features.

Some pioneering work on production of electrolytic manganese has been done in the U.S.A. from low-grade manganese ores by U.S. Bureau of Mines at Boulder City, Nevada, and by the Electromanganese Corporation at Knoxville, Tennessee. Electrolytic manganese is being produced there on a pretty large scale. The metal produced is highly pure. A typical analysis of it is given below :-



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#### **ELECTROLYTIC MANGANESE IN INDIA**

Work on electrolytic production of metallic manganese was taken up by the Standard Metal Company of India at Bombay, and an account of their work was published in 1947. They examined the recovery of manganese from various Indian ores and studied some of the conditions of electrolysis. But the work was not continued further. The National Metallurgical Laboratory, immediately on its establishment, took up the work with a view to evolving a method which could be utilized for commercial production. Central Electro-chemical Laboratory has also reported some work on the subject.

The process developed at National Metallurgical Laboratory<sup>6</sup>,7 can work with almost any grade of ore and finally produce manganese metal of purity 99.95% or better. The manganese ore is ground to fine powder and mixed with saw dust in the ratio  $1:1<sup>s</sup>$  This mixture is then added to the spent liquor containing free acid and the liquor heated with steam under stirring for eight hours. The free acid reacts with the manganese ore reduced by saw dust and pH of the mixture rises. The

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supernatant liquor is run off to a second digesting vessel and charged with only small amount of dioxide manganese ore. By this treatment pH rises enough to cause precipitation of iron and the excess organic matter is destroyed. Arsenic and phosphorous are also eliminated at this stage. Next the decanted liquor is treated with sulphuretted hydrogen to precipitate out any copper, cobalt or nickel. Finally the liquor is adjusted for manganese and ammonium sulphate content and the pH is brought down to 2 and sulphur dioxide gas passed up to a concentration of 0.1 to 0.4 gm per litre. This liquid is then used as feed liquor. Alternatively, the spent liquor is treated with gas reduced manganese ore and subsequently purified as before to obtain feed electrolyte of desired composition. Any of the two methods can be used for regeneration of spent liquor depending upon the facilities available.

Next stage is the electrolysis of the purified electrolyte in suitable cells. Cells as developed in National Metallurgical Laboratory are of lead lined wooden tanks mounted on M.S. frame, fitted with anode diaphragms of canvas. The anodes are made of  $1\%$  silver lead alloy and cathodes are of stainless steel. There is arrangement for continuous feed of fresh electrolyte into cathode chambers and removal of spent liquor from anode chambers. During operation, the cell temperature is regulated by circulating water through lead coils; several anodes and cathodes can be accommodated in a cell and a number of cells can be worked simultaneously for greater output. Fig. 1 shows the cell plant in the semi-pilot plant now in operation at National Metallurgical Laboratory.

The photo shows two cells each accommodating 2 cathodes and 3 anodes. The angle iron frames behind the cells hold the various pipe lines in position. On the high platform purified electrolyte for feeding the cathode chamber is placed along with the cold water reservoir for cooling purposes. The cells are connected to the low tension power line through the switch boards. Empty space on the right is reserved for more cells to he added to the plant. The cells and the plant are designed to run 24 hours a day continuously for days. The output of each of the cells shown in the photo is 7-8 lbs. a day. Table V gives the particulars about the working of the cells.

# TABLE V-DATA ON THE WORKING OF SEMI-PILOT PLANT FOR ELECTROLYTIC MANGANESE





FIG. 1-A *photographic view of the cell plant in the semi-pilot plant at the National Metallurgical Laboratory.* 

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### TABLE VI-COMPOSITION OF MANGANESE METAL PRODUCED IN SEMI-PILOT PLANT



The present capacity of the plant is about 15-16 lb of metal per day. The plant **will** be extended and its capacity doubled in near future.

#### PROSPECTS **OF** ELECTRO-MANGANESE

Technical superiority of electrolytic manganese as a manganese addition agent to steel and nonferrous alloys has been now widely acknowledged. It can be used without any technical difficulty in almost all cases where ferro-manganese is generally added, with definitely equivalent results everywhere and with superior results in majority of the products. The superiority of electrolytic manganese is linked with the high purity of the material. The only impurity is the occluded hydrogen gas which is removed by heating electrolytic manganese at about 500`C under vacuum before its addition to steel. Because of the high purity, no undesirable impurity enters the charge during manganese addition which contamination is likely to happen with any other form of manganese. The possibility of such contamination necessitate scrupulous selection of raw materials and elaborate calculation for making up a charge. These troubles are absent if electrolytic manganese is used. It can also be added in accurately weighed amounts. Storage convenience is another advantage. Extensive tests have been made by U.S. Bureau of Mines regarding use of electrolytic manganese in steel industry. The results of the tests indicate that pure electrolytic manganese can be substituted for any grade of ferro-manganese in electric furnace and open-hearth steels with no disadvantage to quality.

#### LIGHT METAL ALLOYS

Manganese enters into the composition of almost all aluminium and magnesium base alloys. The amount of manganese in such alloys is generally small and can be introduced into the alloy by putting pure manganese oxide or manganese dioxide into the electrolytic bath for aluminium or magnesium. Use of electrolytic manganese in such cases is more convenient and is not likely to introduce impurities. Aluminium alloys containing 1.2-1.5% manganese shows improved corrosion properties and tensile strength.

#### **NEW ALLOYS**

In U.S.A. a number of new alloys are now being produced using electrolytic manganese. Table **VII** gives the composition of the more important of them. With the availability of electrolytic them. With the availability of electrolytic manganese, production of high manganese alloys is **possible. These alloys have important applications in modern scientific and industrial equipment. The high** expansion alloy, containing about 72)% manganese, have found wide application in production of thermostatic bimetals.



It is well known that reserves of copper and zinc ores are very unsatisfactory in India. At the same time our utensils in daily use are made mostly of copper and zinc. Whatever scientific and economic arguments are given in favour of our using utensils made of some other metal, the habit of using brass wares is going to last indefinitely. With the help of electrolytic manganese, it is possible to substitute a part of those two metals in brass. It has been found that if percentage of copper is kept at 65 the remainder can be made up with 30% manganese and the rest with zinc. Such an alloy **will** maintain alpha structure even at a low temperature. A composition of 65% Cu,  $25\%$  Zn and  $10\%$  Mn can be used as a substitute for common brass with significant advantage. This will effect a lot of saving in these metals in short supply. Yet one more application of electrolytic manganese lies in its use in production of substituted stainless steels. It is now wellknown that in the stainless steels of 18 : 8 type the nickel can be replaced by manganese without seriously affecting its rustless properties. These substituted stainless steels have somewhat lower resistance towards acid corrosion. This deficiency can be made up by adding small amounts of molybdenum to **the alloy.** 

**It is, therefore, clear that from technical point of view electrolytic manganese is acceptable in lieu of any other manganese addition agent in almost all metallurgical processes and there is no** technical difficulty in substituting any grade of ferromanganese by electrolytic manganese. It is definitely more advantageous in case of low carbon alloys and non-ferrous alloys. Since a somewhat especial situation obtains **in India in as much as some important minerals are wanting, it is imperative to use manganese, wherever it is possible, in substitution** of the metals the country is deficient in. In India, manganese minerals are an exportable commodity. And she can derive the best advantage

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from her resources by exporting electrolyticmanganese or ferro-manganese instead of raw metallurgical-grade manganese ores. Lastly some solution has to be found for the vast dumps of lowgrade manganese ores which have no buyer in India or outside.

Technically, electrolytic production of manganese from low-grade ores and production of ferromanganese from the metallurgical grade ores will provide adequate solution to the points mentioned earlier. Ore-dressing methods will aid ferromanganese production.

Considering only the financial aspect of these two propositions, production of ferro-manganese is expected to present no special difficulty. Since both electrical energy and manganese ores are available in India, the product should be competitive in price and should be in a position to find market overseas.

The situation is a little more complicated in the case of the electrolytic manganese. At present no electrolytic manganese metal is produced in India and therefore, there is nothing like any figure for cost of production or market price for Indian electrolytic manganese. In U.S.A. the price of electrolytic manganese is about 30 cents per lb. of metal. Calculated in terms of Indian currency it comes to about Rs.  $30/21 = 1.43$ , taking the exchange rate at 21 cents for a rupee. Therefore, price of one long ton of electrolytic manganese comes roughly to slightly over. Rs. 3,000. The standard-grade ferromanganese is now selling at Rs. 1,280 per gross long ton. The difference in prices is very wide and these cost considerations indicate that electrolytic manganese metal cannot be expected to replace standard-grade ferro-manganese. The price of low carbon ferro-manganese is considerably higher and -of the order of Rs. 3,000 per ton in America.

It is worthwhile to consider if the various factors which go to determine the final price of electrolytic manganese are the same in India as in America. There are good grounds to think that the position is better in India. The cost of power, chemicals, and equipment in India will be almost at par with that in America. But the cost of ore and labour will definitely be much lower in India. A preliminary costing done by the National Metallurgical Laboratory shows that cost of production of manganese metal from low-grade ores will be about annas seven per lb. corresponding to Rs. 1,000 per Ion of electrolytic manganese metal. This cost is competitive definitely with that of low carbon ferromanganese and possibly with the price of medium carbon ferro-manganese too. Further, the need for a huge amount of electric power is often a common argument against production of electrolytic manganese metal. It has been shown earlier that already sanctioned schemes for ferro-manganese are going to use electric smelting, and energy consumption per lb. of ferro-manganese is expected to be

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about 1.8 KWH. In electrolytic manganese metal the consumption will be a little over twice this figure but the product will increase considerably in market price. Taking into consideration the peculiarities of Indian mineral resources—for instance high phosphorous in coal, low manganese-iron ratio of manganese ores, and wastage of low-grade ores it seems that production of electrolytic manganese will be a right step towards the country's industrial development.

# PART II—MANGANESE DIOXIDE

Manganese' dioxide plays an important role in dry cell technology, chemical industry, and manufacture of glass and ceramics. In Leclanchétype dry cell it mainly acts as a depolarizor. A very high-grade product containing 85 to 90 per cent manganese dioxide is used for this purpose. Besides the high percentage of manganese dioxide its structure is also very important in determining its cell activity. X-ray diffraction studies $9,10,11$  combined with differential heating curves have revealed the fact that there are five types of manganese dioxide depending upon the source of the natural ore and the method of preparation of the atrificial oxide by electrolytic and different chemical methods $11,12$ . These types are : (i) well-crystallized pyrolusite, (ii) ramsdellite, (iii) cryptomelane, and (iv) delta manganese dioxide believed to be the poorly crystallized cryptomelane. Of these gamma manganese dioxide has been found to be best in electrochemical performance<sup>13</sup>. A Leclanche-type dry cell consists of a zinc container which also serves as a negative pole and a carbon electrode which along with the depolarizing mix forms the positive pole. The depolarizing mix consists of calcined petroleum coke, graphite and manganese dioxide and is impregnated with ammonium chloride and zinc chloride. The exact electrochemical reactions in the dry cell are not yet clearly known and also the part played by manganese dioxide. Extensive research has been carried out since some years prior to the last world war to study the characteristics of manganese dioxide used in dry cells but it has not yet been possible to specify exactly the physical, chemical and other properties on which its depolarizing capacity depends. As a result of these investigations, however, the materials and reactions have been better known. Further dry-cell research is still in progress.

The battery-grade manganese dioxide can be obtained in the natural state or can be synthetically prepared. The most important source of batterygrade ore at the present time is the well-known Nsuta Mine in Gold Coast, West Africa. Different grades of battery ores are now being produced from this mine but most of the exported ores belong to the grade B (86 per cent maneanese dioxide). The

detailed chemical analysis of a typical  $B'$  type ore is : active oxygen as  $MnO<sub>2</sub>$ -86.8%,  $MnO<sub>-3.4%</sub>$ .  $SiO_2-3.2\%, \text{Fe}_2O_3-1.46\%, \text{Al}_2O_3-2.50\%, \text{TiO}_2-$ 0.08%, BaO-0.06%, CaO-0.12%, MgO-0.06%,  $Na<sub>2</sub>O+K<sub>2</sub>O-0.41\%,$  Mo-0.001%, CuO-0.012%,  $Bi_2O_3-0.005\%$ , As<sub>2</sub>O<sub>3</sub>-0.062%, Sb<sub>2</sub>O<sub>3</sub>-0.002%, SnO<sub>2</sub>-0.005%, NiO-0.045%, CoO-0.005%, P<sub>2</sub>O<sub>5</sub>-0.25%,  $No<sub>3</sub> - 0.005$ %. The ore consists chiefly of gamma variety of manganese dioxide with some pyrolusite and cryptomelane.

India is now on the threshold of her industrial development. The main problem of the dry cell industry in India is the difficulty of obtaining the necessary raw materials namely manganese dioxide, graphite, carbon rods, zinc sheets and other chemicals. The growth of national production of dry batteries is now based on expensive imports. It is high time that serious efforts should be made to produce these raw materials in the country. At present there are four companies<sup>14</sup>, namely, National Carbon Company (India) Ltd., Estrella Batteries Ltd., Sunbeam Electrical Industries Ltd., and Solar Batteries and Flashlights Ltd., who manufacture dry batteries in India. Their total rated capacity has been estimated to be about 285 million cells per annum whereas the present requirement is of the order of 320 million cells'''. This demand will increase during the second Five Year Plan period. Due to want of materials, these companies are not able to work at full capacity. India imports more than 5500 tons of manganese dioxide of which more than 4000 tons are used in dry-cell industry. Although the country is richly endowed with manganese deposits—both of the high and low grade—unfortunately the deposit of peroxide ore having battery-active properties is very meagre. Small quantities are available in Keonjhar (Orissa) Shivrajpur (Bombay), Keshapur (Mysore) and Dongri Buzurg (Madhya Pradesh). The exact specification of these battery-grade ores is very difficult to make. Recently, however, Indian Standard Institution<sup>15</sup> has made a brief specification of batterygrade manganese ores of India to serve as a guide for evaluating different grades of ores. Detailed specification, however, warrant further studies including X-ray, electron microscope, and differential thermal analysis. Very small amounts of Indian ores are now being used in dry cells manufactured in India. It is, however, possible to improve the quality of the Indian ore by suitable treatment. Work has already been undertaken by the National Physical Laboratory along this line. It has been found that fairly satisfactory results are obtained by mixing equal amounts of imported and indigenous ores. The difficulty in obtaining raw materials for making dry cells in India can be obviated to a considerable extent by producing the raw materials in the country. Apart from saving foreign exchange it will guard against the period of national emer-

gency. The purpose of this survey is to demonstrate the scope of development of the indiginous commercial production of active manganese dioxide which is one of the strategic materials for the Indian dry-cell industry.

As the natural battery-grade manganese ore is not available in many countries and the demand for dry batteries as easily portable source of, electric supply is constantly increasing, synthetic manganese dioxide is now being used by many battery industries. Japan, Germany, Italy and other countries have developed a number of proprietory processes to produce synthetic active manganese dioxide. These artificial oxides can be produced by different methods—thermal. chemical and electrochemical. A detailed study of the different varieties of manganese dioxide obtained by these methods indicate that electrolytic process gives better product which is mostly gamma manganese dioxide suitable for dry cells. A large volume of work has been done in some countries, especially in U.S.A., to produce synthetic battery-grade manganese dioxide by electrolytic process but the technical details of these processes are mostly covered by patents. The Electrochemical Co. of the United States has evolved a new process<sup>16</sup> by which batterygrade manganese dioxide can be produced on a commercial scale from low-grade domestic ores\_ They are now producing in their plant at Henderson, Nevada, electrolytic battery-active manganese dioxide at the rate of 10 tons per day.

The National Metallurgical Laboratory has already undertaken to produce electrolytic batteryactive manganese dioxide from low-grade Indian ores. Laboratory scale experiments have been successfully completed." Fig. 2 shows the flowsheet of the process developed at the National. Metallurgical Laboratory. The process may briefly be described as given below.

Pure manganese sulphate solution containing about 200 to 250 gms manganese sulphate per litre was prepared from the low grade manganese ores. The ore was crushed, ground to -35 mesh and leached with spent electrolyte containing sulphuric acid. The manganese sulphate solution was then purified by suitable process and after purification it was electrolyzed. The optimum conditions of electrolysis are :—



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FIG. *2—Flow sheet for production of electrolytic manganese dioxide by gas-reduced ore method as ,adopted by Nationa! Metallurgical Laboratory.* 



The current efficiency was 99 per cent and a good deposit of battery-grade manganese dioxide was .obtained. The deposit was grey and hard and easily stripped from the anode. It contained very little lead which was not detrimental to its battery -activity.

India should now undertake producing commercially her requirements of synthetic batterygrade manganese dioxide from low-grade ores. After the successful completion of the laboratory scale experiments, the National Metallurgical Laboratory is going to carry out development-scale experiments in a prototype plant with an output of 100 lb. a day. For the present, to be self-sufficient in battery-grade manganese dioxide, India should produce more than 15 tons of dioxide per day. Initially a 10 ton per day unit may be set up. On the basis of the data available at present, it is possible to make tentative estimates of the cost of construction and operation of a commercial plant of daily capacity 10 tons under Indian conditions. One of the authors (B.C.K.) has visited an electrolytic manganese dioxide plant in U.S.A. and in making this estimate, information collected by him `has been of great help". It is considered necessary to point out that these estimates are not the last

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words but they do indicate the economic feasibility of setting up commercial units for the production of electrolytic manganese dioxide from low quality Indian ores. It may be noted that production of electrolytic manganese dioxide does not require any heavy machinery and equipment. Most of the plant equipment can be fabricated in India from indigenous materials. Also the process developed does not require very close control of operations like electrolytic manganese. On the basis of this estimate a capital outlay of the order of Rs. 35 lakhs is necessary for the construction of a 10 ton/day electrolytic manganese dioxide plant including equipment for (i) crushing and grinding, (ii) roasting, (iii) leaching and purification, (iv) electrolysis, (v) other miscellaneous general equipment and (vi) installation and building. The breakdown of the capital cost is given as follows :—



The cost of operation (recurring cost) including labour, power and supplies per day for a 10/ton plant has been estimated as follows :—





Total processing cost including interest and depreciation but excluding tax and plant insurance etc. is estimated as follows  $:$   $-$ 



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The cost of production of 1 ton of electrolytic manganese dioxide exclusive of taxes, plant insurance, labour insurance, etc. comes to Rs. 458. This cost will decrease as the capacity of the plant increases. Giving a margin for various factors including the cost of equipment, the production cost may be safely put at about Rs. 600 per ton. The present price of imported product is much higher than Rs. 1000 per ton. So it appears that the production of active manganese dioxide from low-grade Indian ores by electrolytic process has great potentialities, apart from the importance that manganese dioxide has as a strategic material for the dry-cell industry in India.

The demand for dry batteries greatly increases during the war to meet defence as well as civilian needs, because a large number of electrical gadgets and instruments for defence purposes are operated by portable source of electric power supplied by batteries. So it is obvious that for national security the country must have the necessary raw materials for the manufacture of dry batteries. The most critical materials for dry-battery production are active manganese dioxide and graphite or carbon black. Both these ingredients are not present in sufficient amounts in India to meet the internal demand. Production of carbon black which is also used in rubber goods, paints and printing inks has already begun in India and two plants are now functioning with a capacity of 2.4 million  $\rm lb.^{19}$  per annum. Other materials, namely, ammonium chloride, zinc chloride, various kinds of card boards and papers etc. are gradually being indigenously produced. So the production of suitable grades of indigenous manganese dioxide which is of major importance should now be taken up and for this purpose the electrolytic process developed at the National Metallurgical Laboratory can be utilized either by the public or the private sector.

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