

UNITED STATES PATENT OFFICE.

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PROCESS FOR THE MANUFACTURE OF CHROMIUM COMPOUNDS.

No Drawing Original application filed March 6, 1926, Serial No. 92,937, and in Germany August 24, 1925.
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I have filed applications in Germany, August 24, 1925, No. F. 59,650 IV/12 m; France, April 15, 1926, application No. 218,535; England, February 8, 1926, application No. 3,553; 5 Czechoslovakia, February 8, 1926, application P. 681; Belgium, April 17, 1926, application No. 264,386; and in Russland, February 24, 1926, application No. 7,159.

The processes for the manufacture of chromium compounds from chrome ore employed in industry up to the present time suffer from the disadvantage that they involve losses of ore which are very difficult to recover. According to most of such processes 10 a diluent is added to the mixture of chrome ore and alkali, as for example chalk, dolomite, iron oxide, etc., these substances being only recoverable in exceptional cases. In most cases they are completely lost with the unattacked ore, thus causing a considerable increase in the cost of the process. Proposals have already been made for using as diluents such materials as can be recovered in a form available for further use. Thus the German 25 Patent No. 166,767 suggests the use of finely crushed chrome ore but the process according to this patent is restricted to the joint use of a hand fed furnace. In order to obtain a satisfactory attack, when working with mechanical furnaces, and with mixtures of finely crushed chrome ore and alkali metal carbonates or hydroxides, very large amounts of ore in proportion to the material used for attacking the ore must be employed, as for 30 example six times the quantity of finely crushed ore. This is due to the fact that a certain amount of diluent material must be present in order to render the melt sufficiently porous, so that the air-oxygen required to bring about the attack can penetrate into the interior of the charge and of the clinker produced.

According to the present invention satisfactory working can be achieved with a much 45 smaller quantity of ore acting as the diluent material and to prevent caking if ore of a definite grain size is employed. In this manner only the most finely crushed portions of the ore are preponderantly attacked while the coarser portions remain practically unaltered and serve as diluent material thus 50 preventing the otherwise unavoidable caking

together of the charge. The application of my invention increases the productive capacity of the whole process concerned. 55

The method of working is as follows:—

The moist or dry mixture of an ore which leaves a residue of 15–30% on a sieve of 70 mesh per cm. with so much of the decomposition agent that the content of the same amounts to 28–32%, is heated in an oxidizing atmosphere to the reaction temperature of over 1100° C. The sintered melt is leached with water, advantageously in the presence of a small amount of acid in order to decompose the aluminate. The chromate which 65 then passes into solution is separated from the residue and worked up in the customary manner.

The residue after the leaching operation 70 consists of unattacked chrome ore, alumina, magnesium and iron-oxide. For the recovery of the unattacked chrome ore mechanical methods may be employed, as for example sedimentation or sorting processes. However it has been discovered that the most advantageous means is not the mechanical but the chemical method, according to which it is possible by treatment of the residue with dilute acid to recover a chrome ore almost 80 completely free from admixed substances. It is essential in this connection, however, that the concentration of the acid should not be too high, as otherwise the final liquors obtained are difficult to filter. Particularly favourable are acids of about 30° Bé. Sulphuric acid of from 5° Bé. to 30° Bé. may be 85 used. It is likewise advantageous to moderately heat the acid although it is advisable not to allow the temperature to rise above 60° C. In general temperatures ranging from atmospheric temperature, 15° C. up to 60° C. may be employed. If these conditions are taken into account a residue is obtained 90 which can be filtered off quite easily. 95

The amount of acid employed may vary within wide limits, either the theoretical amount, an insufficient amount, or an excess, may be employed.

When using the theoretical quantity of acid the final liquors obtained are difficult to filter and this method is therefore only selected in special cases. 100

When an insufficient quantity of acid is em-

played the recovered chrome ore still contains corresponding quantities of iron and magnesium oxides. A proportionate quantity of acid may be added in order to maintain the content of these impurities the same or by varying the acid the amounts of the impurities may be allowed to increase. In the latter case the possibility arises that the melt may become so enriched in iron and magnesium oxides that the decomposition agents added to the charge may just succeed in attacking the whole of the available chrome ore. The resulting residue is then practically free from chromium and may be thrown away.

By the application of an excess of acid pure ore is invariably recovered.

When using either an insufficient quantity of acid or an excess of acid the liquors obtained can be filtered with ease. The velocity of reaction is very great in all cases.

The recovered ore, with or without the addition of fresh ore, is brought to the desired grain size by means of suitable appliances and returned to the charge.

The following examples will serve to illustrate my invention:—

Example 1.—70 parts of ground chrome ore of any composition, leaving a residue of 25% on a sieve of 70 mesh per cm., are intimately mixed with 30 parts of sodium carbonate. According to the construction of the revolving furnace and the feeding arrangement, the mixture may be introduced into the furnace either moist or dry and there heated in the customary manner to above 1100° C. The resulting melt is quenched in water, allowed to soak and the chromium liquor leached and separated from the water-insoluble portion. For the purpose of recovering the unattacked chrome ore, this water-insoluble portion is treated with so much sulfuric acid of 15° Bé. at 50° C. that not all the iron and magnesium oxides are dissolved; the weakly basic liquor is separated off and the residue dried. This resi-

due is mixed with a fresh supply of ore of the required grain size and with sodium carbonate and returned to the furnace. The amount of sulfuric acid required as the process proceeds should be always adjusted so that the basicity of the liquor remains always the same. The yield of chromate obtained is about 90% calculated on the amount of sodium carbonate used.

Example 2.—The water-insoluble portion as obtained in Example 1 after the separation of the chromate liquid is treated with sufficient sulfuric acid of about 20° Bé. in the cold that after the dissolution of all the undesirable impurities a slightly acid solution remains. The heat of reaction leads to a temperature of about 50° C. without special heating. The recovered ore can be employed in a fresh charge without further treatment.

Example 3.—The water-insoluble portion obtained according to Example 1 is treated with sulfuric acid according to the counter-current principle. Thereby the almost extracted ore comes into contact with fresh acid, and thus the acid may be selected of any desired strength. As the process of extraction proceeds further, the most suitable dilution can be easily adjusted by the addition of water.

This application is a division of my application Serial Number 92,937, filed March 6, 1926.

I claim:—

In the manufacture of chromium compounds by heating an excess of chrome ore with an alkaline agent and leaching the chromium compounds out of the reaction mass, the step which comprises preparing the residue after leaching for reuse in the process by treatment with an excess of acid.

In testimony whereof I have hereunto set my hand.

PAUL WEISE.