

# UNITED STATES PATENT OFFICE

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## PRODUCING MOLYBDENUM ORANGE

Arthur Linz, New York, N. Y., and Lynn Wallace Coffey, Detroit, Mich., assignors to Climax Molybdenum Company, New York, N. Y., a corporation of Delaware

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The present invention relates to new and useful improvements in the production of molybdenum orange pigments.

Molybdenum orange pigments are characterized by their enormous covering power, tinting strength, brilliance of color and extreme fastness to light and are now prepared commercially by adding a solution of a soluble bichromate, sulfate and molybdate to a solution of a soluble lead salt. The yellow precipitate which first forms is converted through successive shades of orange to a bright red provided the conditions under which the process is carried out are correct. The properties of the product depend entirely upon these conditions of preparation, with the effect that some of these which are not clearly understood give rise to difficulties in the commercial production of batches which match preceding batches.

It is also commonly supposed that the presence of lead sulphate is necessary to the formation of molybdenum orange despite the fact that Schultze in 1863 (Liebig's Annalen der Chemie (1863) vol. 126, p. 52) showed that it was possible to obtain homogeneous tetragonal crystals of lead molybdate containing up to 42 per cent of lead chromate, which crystals were deep dark red in color in contrast to the lighter red crystals of pure lead chromate.

Objects and advantages of the invention will be set forth in part hereinafter and in part will be obvious herefrom, or may be learned by practice with the invention, the same being realized and attained by means of the steps, instrumentalities and combinations pointed out in the appended claims.

The invention consists in the novel steps, processes, combinations and improvements herein shown and described.

The present invention has for its objects the provision of a novel and improved process for the commercial production of molybdenum orange under reproducible conditions. Another object of the invention is the provision of a novel and improved process for commercially producing a molybdenum orange which has more than usual tinctorial strength, brilliance of color and fastness to light. Still another object of the invention is the provision of a process in which the yield of molybdenum orange is improved.

In accordance with the present invention, molybdenum orange is prepared by mixing a solution of a soluble chromic acid salt and a soluble molybdic acid salt with a solution of a soluble lead salt, and preferably with the addition of a soluble salt forming another insoluble

lead salt, the conditions of mixing being accurately controlled as to acidity, temperature, and preferably being maintained so that there is always an excess of lead in the mixed solution. The additional insoluble lead salt to be formed may be the conventional sulfate, or alternatively may be one or more of many insoluble lead salts such as the borate, perborate, silicate, cyanide, nitroprusside and ferricyanide, chloride and carbonate.

Preferably, the mixed solutions are acid at the time of mixing, are then further acidified to assist in the development of the color and are finally treated with alkali so as to render them very slightly alkaline. After the formation of the molybdenum orange the mixed crystals are stabilized by the formation of a stabilizing substance such as aluminium hydroxide, after which the precipitate is separated and dried, preferably at a moderate or low temperature.

It will be understood that the foregoing general description and the following detailed description as well, are exemplary and explanatory of the invention but are not restrictive thereof.

Referring now in detail to the present preferred and illustrative manner of carrying out the process of the present invention, two solutions are prepared containing the following ingredients in substantially the following proportions:

### Solution #1

Sodium bichromate	
$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ .....	1.15 kg. (3.85 mols)
Sodium molybdate	
$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ .....	.28 kg. (1.1 mols)
Potassium ferricyanide	
$\text{K}_3\text{Fe}(\text{CN})_6$ .....	.394 kg. (1.2 mols)
Sodium hydroxide NaOH....	.32 kg. (8.0 mols)

which are dissolved in 100 liters of water.

### Solution #2

3.47 kg. (10.0 mols+5% excess) lead nitrate dissolved in 100 liters of water.

Other soluble chromic and molybdic acid salts may be employed in suitable proportions in place of the sodium salts, other alkalizing agents may be used and other soluble lead salts may be used in place of the lead nitrate. However, the salts specifically mentioned are preferred for reasons of economy.

The ratio of the molybdic and chromic acid salts specified gives the reddest color, although