This invention relates to a method for the production of diazodinitrophenol and more particularly to a method whereby diazodinitrophenol may be produced directly in a free-flowing form.

Diazodinitrophenol has been known heretofore and methods for its production have been disclosed. Thus, for example, diazodinitrophenol and a method for its production are disclosed in the United States Patents to Dehn Nos. 1,404,687, 1,428,011 and 1,460,708. The compound constitutes a very valuable detonating material for use, for example, in charging blasting caps. However as directly produced by methods heretofore known, it is not free-flowing and hence cannot be readily loaded.

Now, in accordance with this invention, a novel method is provided for the production of diazodinitrophenol and one in its more specific embodiment by which free-flowing diazodinitrophenol may be prepared directly.

Having now indicated in a general way the nature and purpose of this invention, we will proceed to a more detailed description thereof from the broad and from the more specific standpoint.

From the broad standpoint the method embodying this invention involves the diazotization of a salt of picric acid by a salt of nitrous acid and an acid. Thus broadly, this invention involves the diazotization of a picramate by a nitrate and an acid.

More specifically, and especially where the direct production of diazodinitrophenol in a free-flowing form is desired, the acid will be added gradually at a slow rate, or a portion of the acid may be added rapidly at the start with gradual addition of the balance. The rate of addition of the acid, where such is added gradually, following the more specific procedure in accordance with this invention, will depend upon temperature and other variables obtaining during the addition.

In the practical adaptation of this invention any suitable salt of picramic acid, or picramate, may be used. Thus, for example, sodium picramate, ammonium picramate, magnesium picramate, etc., may be used, one being substitutable for the other in equivalent quantities.

Likewise, various salts of nitrous acid, or nitrates, may be used, such, for example, as sodium nitrite, potassium nitrite, barium nitrate, etc., being used in substitution for one another in equivalent quantities.

The acid may be any suitable acid for effecting diazotization of the picramate and nitrite, as, for example, hydrochloric acid or equivalent amounts of sulphuric, phosphoric, acetic, etc. acids.

In carrying out the method the picramate and the nitrates will be in solution or in suspension, or partly in solution and partly in suspension in water. Thus, for example, a suspension of sodium picramate with a concentration as high as 9.7% has been successfully diazotized by the method of this invention without indication that the figure is in any way limiting. The solution or suspension will desirably be substantially neutral, and the concentration of the salts, while they may vary widely, will be such desirably as to give efficient and economic results. The temperature during the diazotization will be controlled and will desirably be maintained, by any suitable means, within about the range 10°-16° C. Where the acid is added gradually for the production of diazodinitrophenol in a free-flowing form, the addition will be at a relatively slow rate, say, for example, at the rate of about 0.001 grams HCl per minute per gram of sodium picramate, or at equivalent rates for other acids and/or picramates. As will be understood, however, the most desirable rate of addition of the acid in any given case will depend upon the temperature and other variables, but will readily be determined in a given case by those skilled in the art. Alternatively to adding all of the acid gradually as a part, say about 25%, may be added rapidly and the balance added gradually.

As illustrative of the practical application of the method in accordance with this invention, for example, about 81.5 pounds (dry weight) of water wet sodium picramate, from which foreign material has been removed, as by screening, say through a 40 mesh screen, is charged into a wooden diazotization tub, fitted with an agitator, about 120 gallons of water. About 19 pounds of sodium nitrite dissolved in 10 gallons of water are then run into the tub and the agitator started. The temperature is adjusted, by any suitable means, for example, by the addition of ice to say about 15° C. About 40 liters of 1:1 hydrochloric acid are then added, preferably at a rate such as to require about 300 minutes for the addition of the acid. During the course of the run the temperature is maintained at about 15°-16° C. by suitable means, as by the addition of ice as required. About five minutes after completion of the addition of the acid the solution is tested with, for example, potassium iodide-starch paper and if a strong positive test is ob-
tained an excess of nitrous acid in the solution
is indicated which shows that the diazotization
of the sodium picramate has been completed.
The addition is then stopped and the charge
dumped, for example, into a suction filter, the
mother liquor drawn off and the diazodinitro-
phenol washed with water to free it from im-
purities.

Following the above procedure the diazo-
dinitrophenol produced will be in free-flowing form
without any subsequent treatment and free-flow-
ing diazodinitrophenol may be obtained by vary-
ing the above procedure to an extent by adding
part of the acid, say for example, 10 liters, quickly
at the start with gradual addition of the balance.
Where the diazodinitrophenol is not desired in a
free-flowing form the acid may be added without
any special attention to the rate of addition.

As has been indicated, the procedure given in
the above illustration by way of example, may be
widely varied without departing from this in-
vention. Likewise, various picramates, nitrites
and acids may be used in varying proportions and
the conditions of diazotization, as temperature,
etc. may be widely varied within operative limits.

As will be obvious, the method in accordance
with this invention is not dependent upon the use
of any particular form of apparatus, means for
cooling, or the like.

What we claim and desire to protect by Letters
Patent is:

1. The method for producing diazodinitro-
phenol in a free flowing form which includes
diazotizing a picramate by the gradual addition
of an acid thereto in the presence of a nitrate and
water.

2. The method for producing diazodinitro-
phenol in a free flowing form which includes
gradually adding an acid to a substantially neu-
tral mixture of a salt of picramic acid and a salt
of nitrous acid and water.

3. The method for producing diazodinitro-
phenol in a free flowing form which includes
diazotizing a picramate by the gradual addition
of an acid thereto in the presence of a nitrate and
water while maintaining the temperature of the
reagents within about the range 10°-16° C.

4. The method for producing diazodinitro-
phenol in a free flowing form which includes
gradually adding an acid to a substantially neu-
tral mixture of sodium picramate and sodium
nitrite in water.

5. The method for producing diazodinitro-
phenol in a free flowing form which includes
gradually adding an acid to a substantially neu-
tral mixture of a salt of picramic acid and a salt
of nitrous acid in water while maintaining the
temperature of the reagents within about the
range 10°-16° C.

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