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METHOD OF EXTRACTING DIPHENYL-AMINE

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4 Claims. (Cl. 260-148)

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amended April 30, 1928; 370 O. G. 757)

The invention described herein may be manu-
factured and used by or for the Government for
governmental purposes, without the payment to
me of any royalty thereon.

5 This invention relates to a method of extract-
ing diphenylamine and its derivatives from
smokeless powder.

Smokeless powder that has deteriorated may
be ground and reworked into new powder after
10 removing any free acid present. If the deter-
iorated powder is to be converted into a product
such as nitrocellulose lacquer it is necessary to
remove the diphenylamine and its nitro-deriva-
tives as well as reduce the viscosity of the nitro-
15 cellulose. It is therefore proposed in the present
invention to provide a simple and economical
method of removing diphenylamine and its de-
rivatives from smokeless powder.

The method consists in grinding deteriorated
20 smokeless powder and treating it with a mixture
of a solvent for diphenylamine which is a gela-
tinizing agent for nitrocellulose and an agent
that prevents or retards gelatinization. An ex-
ample of such a mixture consists of acetone and
25 water, the proportions to be employed depending
upon the completeness of extraction desired, the
length of time of soaking and the number of
washes.

A preferred mixture consisting of 70% to 75%
30 acetone and 25% to 30% water, will give almost
a complete extraction of the original diphenyl-
amine present in the powder. Mixtures of equal
parts of acetone and water have been successfully
employed and slightly reduce the extent of re-
35 moval of the diphenylamine.

The treatment is effected by allowing the com-
minuted powder to soak at room temperature
and with or without agitation in one or more
successive baths of the diluted acetone. The ex-
40 tent of extraction of the diphenylamine is influ-
enced by the fineness of the powder, the number
and length of the washes, and the proportion of
the mixture. Examples are as follows:

In the first two examples given above the wash-
ing was conducted at 60° C., while in the others
room temperature was used.

In no case does the solvent cause the smoke-
less powder to become swollen or adhesive and
no difficulty is experienced in handling the pow-
der during or after extraction.

Another advantage of the process is that it
permits the reduction of the viscosity of the nitro-
cellulose in the powder without the use of a dif-
65 ferent solvent. This can be done by treating the
powder, subsequent to the removal of the di-
phenylamine and its derivatives, with acetone
containing a quantity of acetic acid.

I claim:

1. The method of treating smokeless powder
containing diphenylamine which consists in com-
minuting the powder, and washing it at room
temperature in one or more baths of a mixture
75 composed of acetone 50% to 75% and water
50% to 25%.

2. The method of treating smokeless powder
containing diphenylamine which consists in com-
minuting the powder, and washing it in one or
more baths of a mixture composed of acetone
80 50% to 75% and water 50% to 25%.

3. The method of removing diphenylamine
from comminuted smokeless powder which con-
sists in submitting the powder to the action of a
mixture of acetone 70% to 75% and water 25%
85 to 30%.

4. The method of removing diphenylamine
from comminuted smokeless powder which con-
sists in submitting the powder to the action of a
mixture of acetone 50 to 75 parts and water 50
90 to 25 parts.

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Fineness of powder	Mixture—ace- tone—water		Num- ber of washes	First wash hours	Other wash hours	Total diphenyl- amine left in powder
						Percent
	20	50	50	1	8	0.30
	20	70	30	1	8	0.10
	20	70	30	1	16	0.23
	20	70	30	7	16	0.02
	20	70	30	4	16	0.04
	30	70	30	3	16	0.01
	48	70	30	3	16	0.02