Aug. 9, 1949. M. R. ARNOLD 2,478,619

METHOD OF EXTRACTION

Filed Feb. 16, 1945

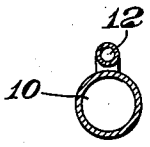


Fig. 2

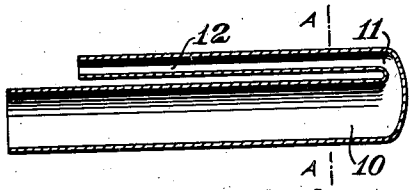


Fig. 1

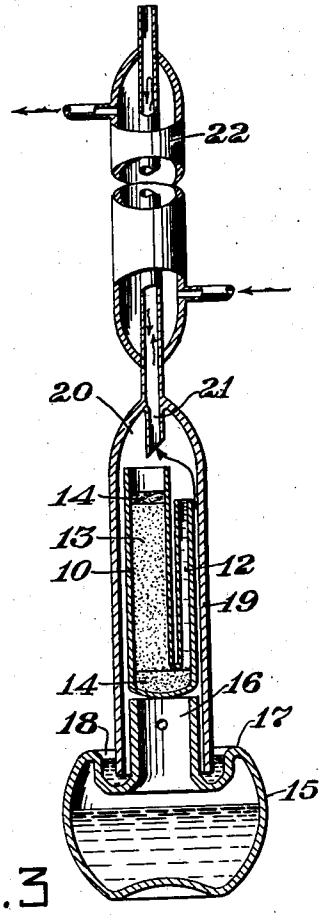


Fig. 3

Melvin R. Arnold INVENTOR.

BY *Fred Aleross*

ATTORNEY

Patented Aug. 9, 1949

2,478,619

UNITED STATES PATENT OFFICE

2,478,619

METHOD OF EXTRACTION

Melvin R. Arnold, Louisville, Ky., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

Application February 16, 1945, Serial No. 578,311

2 Claims. (Cl. 23—312)

1

This invention relates to extraction and more particularly to an improved extraction thimble and method of extraction, which has special application in the ether extraction of colloided nitrocellulose such as smokeless powder, and for other purposes.

This application is a division of my copending application, Ser. No. 479,952, filed March 20, 1943.

The Soxhlet type of extractor operates efficiently when used with substances containing easily extracted material in which the solubility equilibrium is easily obtained, and particularly in non-colloidal material, but it is unsatisfactory when used in the ether extraction of smokeless powder and other forms of colloided nitrocellulose because it permits a periodic exchange of the extraction solvent.

I have now found that these difficulties may be overcome and the time of extraction substantially reduced by means of a new and improved extraction thimble which is described fully hereinafter, together with a preferred embodiment illustrated in detail in the accompanying drawing in which—

Fig. 1 is a horizontal view of an extraction thimble embodying the present invention, and

Fig. 2 is a cross-sectional view taken on line A—A of Fig. 1. Fig. 3 is a cross-sectional view of the improved extraction thimble shown in operative position in the Knorr type apparatus.

Fig. 1 shows the main body 10 of the thimble with the hollow arm 12 communicating with the well of the thimble at the point designated 11. It will be noted that a particular feature of the hollow arm resides in the fact that it terminates at a level substantially below the lip of the thimble so as to maintain constant level of the ether in the thimble at all times during the ether extraction of the sample for analysis contained in the thimble.

The extraction apparatus shown in Fig. 3 comprises the conventional form of Knorr flask 15, having a cylindrical mouth portion or skirt 16, which with the exterior circumferential portion 17 forms a trough or mercury well 18. Seated in this trough is the lower end of an extraction tube 19 adapted to receive the extraction thimble 10 in its upright operating position. The extraction thimble is supported in position in the extraction tube by means of the skirt portion 16 of the flask upon which it rests. The extraction thimble is shown as containing the powder sample 13 having a wadding of cotton or like material 14 at the top and bottom of the sample

2

to maintain the particles of powder in the thimble during extraction.

The upper portion of the extraction tube forms a vapor chamber 20 communicating with the condensing tube 21 which is surrounded by the condenser shell or cooling jacket 22.

As will be noted from the illustration in Fig. 3, a full column of liquid is maintained in the side arm 12 of the extraction thimble at all times during extraction so as to maintain a constant level of ether over the sample of powder in the thimble while the vapors from the heated flask 15 move upwardly through the extraction tube 19.

To accomplish the best results it is preferable that the sample of smokeless powder or other form of nitrocellulose intended for analysis be comminuted, especially if the grains or particles are of substantial size.

Compared with the straight type thimble requiring 48 hours' extraction time for the extraction of ether soluble material from a sample of smokeless powder, the extraction time can be completed in 24 hours by means of my new and improved thimble, and in most cases this time can be reduced to within 18 to 20 hours. This reduced period of extraction time is made possible by the fact that the colloided material is maintained completely immersed in ether held in the thimble throughout the entire extraction period. This reduced time of extraction is an important desideratum in the control of manufacture of smokeless powder or other forms of colloided nitrocellulose in large scale production.

The foregoing is given by way of illustration of a specific embodiment of my invention and is not to be taken in any manner as a limitation of the invention.

It will be apparent from the foregoing detailed description that this improved extraction thimble and method of extraction will be found useful not only in the analyses of smokeless powder samples, but may also be used in determining the total soluble extract in other materials containing extractable matter. While ether is considered preferable for extracting soluble material from smokeless powder, this improved thimble may be used equally well with other solvents.

Apart from accomplishing its purpose in giving greatly improved results over the well-known Soxhlet extractor, it is free from the undesirable properties of fragility and high cost found in such prior known devices. My new and improved thimble is adapted to be and preferably

is used with the Knorr type of condenser and flask.

I claim:

1. The method of extracting ether-soluble material from insoluble nitrocellulose which comprises continuously dissolving the ether soluble material by means of a solvent, continuously removing the dissolved material from the insoluble nitrocellulose while retaining substantially all of the solvent and dissolved material within the extraction system, and continuously recycling the solvent and effecting contact of said recycled solvent by gravity flow with the material to be treated, while maintaining constant immersion of the insoluble nitrocellulose in the solvent throughout the extraction period.

2. The method of extracting soluble material from smokeless powder which comprises continuously dissolving soluble material by means of a solvent, continuously removing dissolved material from the insoluble nitrocellulose while retaining substantially all of the solvent and dissolved material within the extraction system, and continuously recycling the solvent and effecting contact of said recycled solvent by gravity flow with the material to be treated, while maintaining constant immersion of the smokeless powder in the solvent throughout the extraction period.

MELVIN R. ARNOLD.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,538,574	Mittasch et al.	May 19, 1925
1,942,848	Taylor	Jan. 9, 1934
2,058,300	Cramer	Oct. 20, 1936
2,377,135	Dinley	May 29, 1945