

1073.896

PATENT SPECIFICATION

DRAWINGS ATTACHED

1073.896



Date of Application and filing Complete Specification: May 7, 1965.
No. 19430/65.

Application made in United States of America (No. 367446) on May 14, 1964.
Complete Specification Published: June 28, 1967.

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Index at acceptance:—A2 C(1E2, 1E3)

Int. Cl.:—A 24 c 5/50

COMPLETE SPECIFICATION

Tobacco Smoke Filter

We, PHILIP MORRIS INCORPORATED, a corporation organised under the laws of the State of Virginia, United States of America, of 100 Park Avenue, New York 17, State of New York, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to tobacco smoke filters comprising synthetic fibers, activated carbon and a high molecular weight polyalkylene glycol. Synthetic fibers, for example, cellulose esters, have found widespread use in tobacco smoke filters due to the ease with which they can be manufactured into filter rods on standard cigarette manufacturing equipment. These synthetic fibers generally comprise cellulose acetate in the form of crimped, continuous fibers or filaments. Filters made of cellulose ester fibers function, in general, by removing a portion of the particulate matter from the smoke which passes through the fibers. The crimping or other physical positioning of the fibers within the filter serves to increase the surface area of the filaments which come in contact with the smoke. However, filters consisting of such fibers alone do not remove any significant amounts of undesirable gas phase constituents from the tobacco smoke.

Certain materials have also been applied to synthetic fibers which are used in filters in order to make the fibers more effective in picking up the particulate matter in the smoke. Such materials may, for example, be starch, ground cellulose or the like and are generally held in place by an adhesive or plasticizer such as dimethoxyethylphthalate, methylphthalylethylglycollate or triacetin. However, these materials do not materially alter the failure of the fibers to remove un-

desirable gas phase constituents from the smoke.

Other materials which are, in effect, self-bonding materials have been suggested for incorporation in synthetic fibers to be used in tobacco smoke filters. These materials include, for example, certain types of wax particles, such as paraffin or polyethylene wax, and certain sugar esters, such as sucrose acetate propionate. However, filters containing such additives also fail to remove any substantial amount of the undesirable elements which are present in the gas phase of the tobacco smoke.

Certain adsorbent particles, as exemplified by activated carbon, have also been employed in tobacco smoke filters. Such adsorbent particles have been employed in conjunction with crimped paper and serve to increase the effectiveness of such filters for removing the particulate matter in the smoke, as well as functioning to remove undesirable gas phase constituents from the smoke. However, when activated carbon has been used in this manner, it has been employed in the form of very fine particles or dust, because it had been found that larger carbon particles are not completely satisfactory when incorporated with paper. The larger particles have been found to be virtually impossible to employ in paper-type filters.

Since paper-type filters have been found to be less efficient than synthetic fiber or tow-type filters, activated carbon particles have also been suggested for incorporation in synthetic filaments to form tobacco smoke filters. However, it has been generally recognized that the particles of carbon will not by themselves adhere well to the synthetic fibers in the filter and adhesives such as triacetin, dimethoxyethylphthalate or methylphthalylethylglycollate have been employed to hold the particles on the fibers. Such adhesives have been found to poison or inactivate the activated carbon to a considerable extent.

[Price 4s. 6d.]

Some of the disadvantages of incorporating carbon particles in synthetic filaments have been overcome by the use of an improved adhesive mixture comprising polyvinyl pyrrolidone and a polyhydric alcohol, such as glycerol. Such an adhesive has made it possible to employ carbon particles over a wider range of sizes than had previously been possible. However, even such an improved adhesive has had only limited effectiveness and it has been taught that for such applications the carbon particles should preferably be of such a size that no more than a very small percentage of them (most preferably no more than 5%) are larger than 48 mesh.

While it has also been proposed to employ larger particles of carbon in a filter, such particles have not been used in the fiber-containing section. Instead, such particles have been used in a separate filter section comprised solely of the carbon particles and this section has been placed between two non-carbon containing filter sections. This type of filter has an advantage over the previous filters in that the carbon particles need not be contacted with an adhesive to hold them on paper or tow. However, such filters create a different problem; in that the loose carbon particles tend to migrate from the section in which they are placed to other parts of the cigarette. Furthermore, this type of filter has the disadvantage that the carbon particles do not contact all of the tobacco smoke to the same degree. In such a filter, there is some settling of the carbon particles within the carbon-containing section of the filter during storage and transit of the cigarettes, creating an open space above the carbon. There is also a considerable amount of empty space around each of the carbon particles. Thus, a substantial percentage of the tobacco smoke, following a path of least resistance, can pass through these empty spaces in the filter without any significant contact between the smoke and the carbon. As will be more fully set forth below, the present invention overcomes this disadvantage and provides for intimate contact between substantially all of the tobacco smoke and the carbon particles.

Filters have also been made with adhesives to adhere the carbon particles together to form a disk or plug composed of carbon and adhesive. While such construction tends to overcome the problem of carbon particle migration, the adhesives in such filters have been found to have been employed in such a manner that the carbon is at least partially deactivated by the adhesives. Furthermore, such filters have also been found to give relatively poor contact between the smoke and the carbon particles.

In one aspect the invention provides a filter for tobacco smoke comprising synthetic filaments or fibres, activated carbon particles, particularly activated carbon particles of rela-

tively large size, and a high molecular weight polyalkylene glycol (as hereinafter defined).

The high molecular weight polyalkylene glycol, which will be described in more detail hereinafter, serves to selectively remove certain gaseous phase components from the tobacco smoke as well as to bind the carbon particles to the filtering medium without adversely affecting the character or the quality of the smoke which passes through the filter. In addition, the high molecular weight polyalkylene glycols are compatible with the materials generally employed in filter manufacture and do not have any objectionable taste or odour.

The invention is illustrated in the accompanying drawings wherein.

Fig. 1 is a longitudinal sectional view of a cigarette embodying the filter of the present invention.

Fig. 2 is a cross-sectional view of the cigarette shown in Fig. 1, taken along the lines 2—2.

Fig. 3 is a longitudinal sectional view of a modification of the present invention.

Fig. 4 is a cross-sectional view of the cigarette shown in Fig. 3, taken along the lines 4—4.

Fig. 5 is a magnified view of a portion of the cigarette shown in Figs. 1 and 2, showing in greater detail the body of filtering material prepared in accordance with the present invention.

Referring to Figs. 1 and 2, there is shown a cigarette 1 having a paper cylinder 2. Shredded tobacco 3 is contained within paper cylinder 2. Paper cylinder 4 is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder 2. Paper cylinder 4 generally defines the filter unit of cigarette 1. Paper cylinder 4 contains a carbon-free filter section 5, which is preferably tow, which is contained within paper cylinder 6. Carbon-containing section 7 contained within paper cylinder 8 comprises cellulose acetate filaments 9 and carbon particles 10 which are held in place by high molecular weight polyethylene glycol adhesive 11. The filter unit, which is defined by paper cylinder 4, is joined to the filler section, which is defined by paper cylinder 2, by paper cylinder 15, which covers all of paper cylinder 4 and a portion of paper cylinder 2.

Figs. 3 and 4 illustrate another form of the present invention wherein there is shown a cigarette 10 having a paper cylinder 20. Shredded tobacco 30 is contained within paper cylinder 20. Paper cylinder 40 is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder 20. Paper cylinder 40 generally defines the filter unit of cigarette 10. Paper cylinder 40 contains a carbon-free filter section 50, which is preferably tow, which is contained within paper cylinder 60. Carbon-

containing section 70 contained within paper cylinder 80 comprises cellulose acetate filaments 90 and carbon particles 100 which are held in place by high molecular weight polyethylene glycol adhesive 110. Carbon-free section 55 contained within paper cylinder 56 is positioned between section 70 and filler 30 and serves to isolate the carbon-containing section from the filler as well as to provide additional particulate matter filtration. The filter unit, which is defined by paper cylinder 40, is joined to the filler section, which is defined by paper cylinder 20, by paper cylinder 150, which covers all of paper cylinder 40 and a portion of paper cylinder 20.

With particular reference to Fig. 5, which illustrates in detail a preferred form of the present invention, cellulose acetate fibers 9 are shown as being substantially continuous, crimped filter elements which are substantially longitudinally aligned and spaced to provide passage for tobacco smoke therethrough. Relatively large carbon particles 10 are positioned within the spaces between fibers 9 and are held in position by high molecular weight polyethylene glycol adhesive 11.

It will be seen from Fig. 5 that each particle of carbon generally extends within several of the channel-like spaces which are formed by the cellulose acetate filaments. These channel-like spaces generally define a plurality of tortuous paths along which the tobacco smoke travels. During such travel, the tobacco smoke encounters the relatively large particles and is diverted thereby through adjacent passages. In this manner, the smoke not only passes around each particle with substantial contact occurring but also is forced through a more complex passage through the filaments themselves. Thus, the smoke is forced into more intimate contact with both the carbon particles and cellulose acetate fibers than has heretofore been possible.

It will also be noted from Fig. 5 that some of the particles of high molecular weight polyethylene glycol adhere to the cellulose acetate fibers as distinct particles and that such particles do not hold any carbon in place by direct contact with the carbon. These particles also serve to enhance the filtering ability of such a filter, since they provide additional barriers to the passage through the filter of the particulate matter in the smoke. In addition, the high molecular weight polyethylene glycol itself serves to remove phenols and other undesirable gas-phase constituents from the tobacco smoke.

It will be understood that the materials employed in the cigarettes shown in the drawing is merely illustrative. For example, the form of carbon particles shown and the specific polyalkylene glycols described are merely representative of the carbon particles and polyalkylene glycols which may in general be used. Likewise the paper coverings for the

respective filter sections may also be varied from the arrangements shown in the drawing, dependent upon various factors, such as manufacturing convenience.

In order to prepare the tobacco smoke filter of the present invention, the cellulose acetate fibers or similar fibers are preferably spread out and thereafter sprayed with a mist containing the high molecular weight polyalkylene glycol. In this manner there is formed on the fibers a non-continuous covering comprising small particles of the high molecular weight polyalkylene glycol.

The high molecular weight polyalkylene glycol may be sprayed or applied to the fibers by means of a centrifugal applicator. In order to accomplish this, the high molecular weight polyalkylene glycol must be in a flowable form. This can be accomplished by heating the high molecular weight polyalkylene glycol to a temperature slightly higher than its melting point. It is then fed to a rotating perforated cylinder, from which it is applied to the fibers in the form of a fine mist. Alternatively, it may be sprayed by spray equipment. It may also be dissolved in water and applied as a solution, if desired, or by other suitable means.

While the above method for applying the high molecular weight polyalkylene glycol is preferred, it may also be deposited on the fibers as a film, for example, a continuous or semi-continuous coating.

For convenience, the fibers after the high molecular weight polyalkylene glycol has been applied will be referred to as "coated fibers", regardless of whether the glycol was applied by centrifugal applicator, by spraying or otherwise, and regardless of whether the glycol is applied as discrete particles, as a non-continuous coating or as a continuous coating.

After the application of the high molecular weight polyalkylene glycol, the carbon particles are applied to the coated fibers.

Some of the high molecular weight polyalkylene glycols which may be employed will not completely harden after being applied to the fibers and will remain on the fibers, for example, as extremely viscous liquids. In this case their adhesive action in that state is such that the carbon can be added either immediately after the coating is applied or at a later time, even after standing at room temperature.

High molecular weight polyalkylene glycols, which must be heated to make them flowable, will, upon cooling, harden after application to the fibers. In this case, the activated carbon particles should be dispersed over the coated fibers before the high molecular weight polyalkylene glycol particles have solidified on the fibers. Obviously, the coated fibers can, where necessary, be maintained or placed in an atmosphere which is at a sufficiently high temperature so that the high molecular weight polyalkylene glycols possess tacky or adhesive

qualities when the carbon particles are applied.

The carbon-containing coated fibers, still before the polyalkylene glycol has solidified, may then be gathered together into the desired configuration for a filter and the resulting material formed within a paper cylinder or other wrapper to be held in the desired shape. The forming operations may be those conventionally employed in the manufacture of cigarette filters.

The synthetic fibers which may be employed in accordance with the present invention are preferably cellulose acetate fibers and may be cellulose acetate tow or yarn having a total denier of from about 25,000 to about 75,000, and preferably from about 30,000 to about 50,000, and having a denier per filament of from about 1 to about 16, and preferably from about 3 to about 6. Preferably, the cellulose acetate fibers or filaments are generally longitudinally aligned and coextensive with the length of the filter element and have portions thereof which are crimped into a different alignment from the general orientation of the filaments, whereby the filaments have certain portions which somewhat intermesh, as illustrated in the drawing. While cellulose acetate fibers are the preferred materials, other cellulose esters, for example, cellulose butyrate, or other types of synthetic fibers and mixtures of synthetic fibers may also be employed as the fibers of the present invention.

The activated carbon particles which are

employed in accordance with the present invention may have a particle size of from about 10 to about 325 mesh (United States Standard) but preferably are relatively large particles of from 10 to 45 mesh size, i.e., substantially all of the particles will pass through a 10 mesh screen and will be retained on a 45 mesh screen. Most preferably, at least 95% of the particles should be retained on a 30 mesh screen and should pass through a 12 mesh screen. The particles may be roughly spherical in shape or may have other regular or irregular shapes, depending on how they are produced. Preferably, they have irregular shapes, such as are produced by grinding coconut-type carbon.

The activated carbon which is employed may be one of various known types, such as coke base carbon or nutshell carbon, or any other suitable carbon.

Preferably, the carbon employed is a coconut-type carbon. Such carbons have a somewhat irregular shape, and are therefore held somewhat more easily in position within the mass of synthetic filaments. They also afford good contact and good pick up of undesirable gas phase constituents.

A particularly preferred carbon is a coconut carbon such as the PCB grade activated charcoal manufactured by Pittsburgh Coke and Chemical Company. This carbon has a high rate of adsorption and a high retentivity. As one specific example, the carbon may have the following properties:

70	Physical Properties	
	Total Surface Area Sq. Meters/Gram	1200
	Apparent Density	27.5 lbs./Ft. ³
	Pore Volume (within Particle)	+9.4 CC/Gm.
	Voids in Dense Packed Column %	38—40
75	Specifications	
	Iodine No. Mg./Gram	1200 Min.
	Carbon Tetrachloride Adsorption Wt. %	60% Min.
	Ash—Maximum %	4%
	Moisture—Max. % as Packed	3%
80	Hardness Number, Min.	92 Min.

Size Breakdown—12×30 Mesh

	Sieve No.	% Retained
85	+12	0—5
	12×16	20—40
	16×20	40—85
	20×30	10—20
	—30	0—5

The high molecular weight polyalkylene glycol may be polyethylene glycols, polypropylene glycols, glycols containing both $-C_2H_4O-$ and $-C_3H_6O-$ groups and may also be mono- or di-esters of such high molecular weight polyalkylene glycols, for example, the methoxy esters of polyethylene

glycols. As used herein, the term "high molecular weight polyalkylene glycol" is intended to include all such materials, including the esters, having molecular weights of from about 600 to about 20,000.

Illustrative of such materials are polyethylene glycols having the general formula:



wherein x is an integer having a value of from about 12 to about 453. Such materials may be prepared by conventional methods. For example, 2 moles of the monosodium salt of diethylene glycol of beta, beta'-dichloroethyl ether can be condensed to yield hexaethylene

glycol dichloride, which is then condensed with 2 moles of the monopotassium salt of hexaethylene glycol to yield an 18 membered compound. The synthesis may be continued in this manner to yield even high molecular weight compounds.

Examples of suitable polyalkylene glycols include materials sold under the trade name Carbowax. The carbowax polyethylene glycols are commonly referred to in abbreviated form. For example, Carbowax 600 is the abbreviated form for Carbowax brand polyethylene glycol 600.

Carbowax 600 is a preferred adhesive material. This material has an average molecular weight of about 600 and is a soft, white, waxy substance at room temperature.

Illustrative of other Carbowax materials which may be employed are: Carbowax 1000, Carbowax 1500 W, Carbowax 1540, Carbowax 4000 and Carbowax 6000. These materials have the following properties:

Carbowax 1000. Average molecular weight 950—1050. Soft, white, wax solid. Solidifying range 35—40°C. Flash pt. >450°F. Saybolt viscosity at 210°F., sec. 70 to 100. Solubility in water at 20° approx. 70% w/w. Comparative hygroscopicity=5 (glycerol=100).

Carbowax 1500 W. Average molecular weight 500 to 600. Soft, white, waxy solid having the consistency of low-melting petrolatum. Specific gravity at 20°C (relative to water at 20°C) 1.151. Solidifying range 35 to 40°C. Flash pt. 430°F. Solubility in water at 20° approx. 73% w/w. Completely soluble in water at 50°C. Insoluble in petr. ether. The pH of a 5% aq. soln. is about 4.6. Comparative hygroscopicity=30 (glycerol=100). Saybolt viscosity at 210°F., sec. 60 to 90. LD₅₀ i.p. in mice: 9.2 g./kg.

Carbowax 1540. Average molecular weight 1300—1600. Soft, white, waxy solid. Specific gravity at 20°C (relative to water at 20°C) 1.15. Solidifying range 40—50°C. Flash pt. >450°F. Solubility in water at 20°C approx. 70% w/w. Insoluble in petroleum ether. The pH of a 5% aq. soln. is about 6.5. Comparative hygroscopicity=5 (glycerol=100). Saybolt viscosity at 210°F., sec. 100 to 150.

Carbowax 4000. Average molecular weight 3000 to 3700. Hard, white, waxy solid. Specific gravity at 20°C (relative to water at 20°C) 1.204. Solidifying range 50—55°C. Flash pt. 475°F. Solubility in water at 20°C approx. 62%. Insoluble in petroleum ether. The pH of a 5% aq. soln. is about 6.35. Comparative hygroscopicity=1 (glycerol=100). Saybolt viscosity at 210°F., sec. 400 to 800.

Carbowax 6000. Average molecular weight 6000—7500. Hard, white, waxy solid. Solidifying range 58—62°C. Flash pt. >475°F. Solubility in water at 20°C approx. 50% w/

w. Insoluble in petr. ether. Comparative hygroscopicity=1 (glycerol=100). Saybolt viscosity at 210°F., sec. 6000—7500.

Alkoxy polyethylene glycols may also be employed, for example, methoxy polyethylene glycol or other lower alkoxy polyethylene glycols. A specific illustration of such material, and a preferred material in this invention, is Carbowax methoxy polyethylene glycol 750 which has the following properties: Average molecular weight of 715—785; specific gravity at 40/20°C. of 1.094; viscosity CKS at 210°F. of 10.5 and freezing range of 27 to 32°C. This material has been found to give even better results than Carbowax 600.

High molecular weight polypropylene glycols and mixed glycols having similar properties to the high molecular weight polyethylene glycols may also be employed.

The Carbowaxes are more completely described in the publication of Union Carbide Chemicals Company entitled "Carbowax Polyethylene Glycols".

In filter plugs which embody the present invention, from about 2 to 200 parts, by weight, of carbon and from about 2 to 25 parts by weight of high molecular weight polyalkylene glycol are employed per 100 parts of fiber. Preferably, when the filter plug is to be employed in a cigarette or similar product, sufficient carbon should be present to provide from about 2 to about 150 mg. of carbon per cigarette. Most preferably, the amount of carbon is about equal, for example, no more than 20%, by weight, higher or lower, to the combined weight of tow, paper and paste employed in the filter plug. The amount of high molecular weight polyalkylene glycol employed, under such circumstances, will preferably be about 0.1% to about 0.3%, by weight, of the amount of carbon employed.

Filter plugs embodying the present invention may contain other additives, for example other adsorbents, such as silica gel, rice starch, activated aluminas and the like, but preferably contains the high molecular weight polyalkylene glycol as the sole adhesive or binding agent.

Filter plugs embodying the present invention will generally be from about 4 to 25 mm. in length, when employed in cigarettes or the like. The plugs are preferably incorporated in combination with an outer carbon-free plug, which may be a paper type plug or a tow type plug. Preferably, the outer plug is a cellulose acetate crimped fiber type plug having a total denier of from about 25,000 to 100,000, a denier per fiber of from about 1 to 16, a plug length of from about 4 to 20 mm. and a maximum RTD (resistance to draw) of about 6 inches of water.

An inner plug positioned between the tobacco and the carbon-containing plug may also be employed, if desired. This inner plug

may be similar in nature to the outer plug.

Both the inner plug and the outer plug, when employed, may have incorporated therein high molecular weight polyethylene glycols, triacetin, various waxes, and other additives and similar materials and may also include various flavorants, if desired.

It is to be understood, however, that when an inner plug and an outer plug are employed in combination with a carbon-containing filter of the present invention, the inner and outer plugs need not be of the same construction or contain the same additives, if any. Thus, for example, the outer plug may contain various flavorants which are not present in the inner plug.

For a further understanding of the present invention, reference may be had to the following examples, which merely serve to illustrate certain forms of the present invention.

EXAMPLE 1

A 5 denier per filament cellulose acetate yarn in the form of crimped tow containing 40,000 filaments was pulled over a series of air jets which spread the filaments over a width of about 8—12 inches. While in this spread-out condition, the filaments were passed through a centrifugal applicator wherein a high molecular weight polyethylene glycol (Carbowax 600) which has been maintained in a reservoir at a temperature of from 30 to 35°C. (i.e., about 5—10°C. above its melting point) is sprayed on both the upper and lower surfaces of the spread-out tow maintained at about the same temperature as the Carbowax 600. Other high molecular weight polyalkylene glycols are also preferably applied to tow, in accordance with the invention, at a temperature slightly above their melting point, for example, about 10°C. above their melting point.

The high molecular weight polyethylene glycol is applied to the fibers in the form of small droplets. The resulting coated spread-out tow is then passed beneath a hopper through which particles of PCB type carbon, having a particle size of 12 to 30 mesh are passed, whereby the carbon particles are uniformly distributed across the surface of the spread-out and coated fibers. The carbon particles are fed by means of a standard magnet vibratory feed unit. The resulting material is then fed through a plenum chamber and is thereafter passed through a device whereby the tow is formed into a cylindrical shape and is wrapped with a paper wrapper to produce a continuous rod with a circumference of about 25 mm.

The resulting rod is then ultimately cut into 12.5/ mm. filter tip lengths which gradually become firm due to the adhesive action of the high molecular weight polyethylene glycol on the cellulose acetate fibers and on the carbon particles. Analyses of this filter

section from which the paper wrapper was removed show that it consisted of about 48% cellulose acetate filaments, 3% high molecular weight polyethylene glycol and 49% activated carbon.

The activated carbon was not readily dislodged from the fibers even with vigorous tapping of the filter section against a hard surface.

Examination of a number of such filter sections indicated that the carbon particles extend in general into more than one of the paths formed by the cellulose acetate filaments and that the carbon particles were held in place by means of the Carbowax 600, particles of which were also deposited on the filaments apart from the carbon particles. The Carbowax 600 served to hold the carbon particles in place by two actions: (1) it bound the cellulose acetate filaments together to form a web which held the carbon particles firmly in place, the size and shape of the particular coconut carbon employed facilitating this holding action and (2) it directly bound carbon particles to the fibers by adhesive action.

Ten of these filter tips were each separately attached to ten outer filter plugs of 4.5 denier cellulose acetate tow containing 68,000 filaments. These outer plugs were 7.5 mm. long and contained 10% of a plasticizer comprising 50% Carbowax 600 and 50% triacetin. The two filter sections, in each case, were attached to a standard tobacco mixture contained in a length of cigarette paper sufficient to give an over-all cigarette length of 85 mm. The outer plug also contained selected flavorants.

The cigarettes were found to have the following properties (based on an average of the ten cigarettes):

- (1) Filter efficiency (% tar removed) 50.1%
- (2) Total RTD 4.56 inches of water
- (3) Inner plug RTD 2.57 inches of water
- (4) Outer plug RTD 1.09 inches of water

A commercially available filter cigarette of the cellulose acetate type and a commercially available cigarette having a filter section comprised of free particles of carbon were tested in comparison with the cigarettes prepared in accordance with this invention with the following results: the two commercially available cigarettes had lower efficiencies (41.4 and 46.4%, respectively) than the present cigarettes and, when tested by chromatographic determinations for the removal of gas phase components, the present filters were found to remove from the smoke greater amounts of most of the undesirable gas phase constituents, such as acetaldehyde, acrolein, methyl ethyl ketone, acetonitrile, benzene and the like.

Cigarettes were prepared in substantially the same manner, employing (separately) in place of Carbowax 600, Carbowax 750, Carbowax 1000 and Carbowax 1540, with similar results to those obtained using Carbowax 600. Be-

cause of their more solid consistency, these higher molecular weight materials are preferred over the Carbowax 600.

EXAMPLE 2

5 Cigarettes prepared in a similar manner to Example 1 were prepared in two batches: one batch had carbon plugs containing the same amount of Carbowax 600 as the cigarette of Example 1 and the second batch had
10 carbon plugs containing no Carbowax 600. Except for the Carbowax 600, the cigarettes were substantially identical. Smoke from these cigarettes was analyzed. It was found that the Carbowax-containing cigarettes removed about
15 18% more phenols than did the Carbowax-free cigarettes, with all other efficiencies and removals of the cigarettes tested being approximately the same for each type of cigarette.

20 Resistance to draw, also referred to in this specification as RTD, was determined as follows:

A vacuum system was set to pull an air flow of 1050 cc./min. by inserting the tapered end of a standard capillary tube through the dental dam of the cigarette holder and adjusting the reading on the water manometer to the correct RTD. The water level of the manometer was set at zero before inserting the standard
30 capillary.

Then, the butt end of a cigarette or plug was inserted to a depth of 5 mm. in the dental dam of the cigarette holder. The pressure drop behind this cigarette with 1050
35 cc./min. of air flow passing through was read directly as RTD (inches water) from the inclined water manometer.

The molecular weight described in this specification is an average molecular weight
40 and is determined as follows:

(1) Preparation of phthalic anhydride-pyridine reagent: Add 42 g. of phthalic anhydride to 300 ml. of freshly distilled pyridine

contained in a one-quart brown glass-stoppered bottle. Shake the bottle vigorously until complete solution is effected. The reagent preferably should stand overnight before using; however, the solution may be heated under hot tap water until a slight cooling of the reagent occurs, indicating complete reaction.

(2) Procedure: Prepare a sufficient number of clean dry heat-resistant pressure bottles to make all control and sample determinations in duplicate. Carefully pipet 25 ml. of the phthalic anhydride-pyridine reagent into each of the bottles, using the same pipet for each transfer. Reserve two of the bottles as blanks.

Into each of the other bottles introduce the specified amount of sample in the manner indicated. Make all weighings to the nearest 0.1 mg.

Fit the bottles with pressure stoppers and wrap each securely in a canvas bag. Place the samples and blanks as close together as possible in a water bath maintained at $98 \pm 2^\circ\text{C}$. for 30 minutes. Maintain sufficient water in the bath to just cover the liquid in the bottles. Remove the bottles from the bath and allow them to cool in air to room temperature. Do not remove the wrappers from the bottles while they are hot, or attempt to hasten the cooling by immersing them in cold water, as a serious accident can result from the breakage of the bottles. When the bottles have cooled, loosen the wrappers, uncap to release any pressure, and then remove the wrappers.

To each bottle add exactly 50 ml. of standard 0.5 N sodium hydroxide, using the same pipet for each addition. Be sure to allow the same drainage time for each bottle as this amount is not considered in the final calculation. Add 5 drops of a 1.0 per cent pyridine solution of phenolphthalein indicator and titrate with standard 0.5 N sodium hydroxide to a pink endpoint permanent for at least 15 seconds.

(3) Calculations:

$$\frac{\text{grams sample} \times 200}{(B-A)N} = \text{average molecular weight}$$

90 A=ml. of N normal NaOH required for the sample

B=average ml. of N normal NaOH required for the control

WHAT WE CLAIM IS:—

95 1. A filter for tobacco smoke comprising synthetic filaments or fibres, activated carbon particles and a high molecular weight polyalkylene glycol (as hereinbefore defined).

2. A filter according to claim 1, comprising synthetic filaments which are at least in part
100 cellulose acetate.

3. A filter according to claim 2, wherein

the cellulose acetate filaments each have a denier of from 3 to 6.

4. A filter according to any preceding claim wherein the activated carbon is coconut carbon.
105

5. A filter according to any preceding claim wherein the activated carbon particles have a U.S. standard mesh size of from 10 to 44.

6. A filter according to claim 5, wherein the size of the activated carbon particles is such that 95% are retained on a 30 U.S. standard mesh screen and 95% pass through a 12 U.S. standard mesh screen.
110

7. A filter according to any preceding claim, wherein the high molecular weight poly-
115



2000



cause of their more solid consistency, these higher molecular weight materials are preferred over the Carbowax 600.

EXAMPLE 2

5 Cigarettes prepared in a similar manner to Example 1 were prepared in two batches: one batch had carbon plugs containing the same amount of Carbowax 600 as the cigarette of Example 1 and the second batch had
10 carbon plugs containing no Carbowax 600. Except for the Carbowax 600, the cigarettes were substantially identical. Smoke from these cigarettes was analyzed. It was found that the Carbowax-containing cigarettes removed about
15 18% more phenols than did the Carbowax-free cigarettes, with all other efficiencies and removals of the cigarettes tested being approximately the same for each type of cigarette.

20 Resistance to draw, also referred to in this specification as RTD, was determined as follows:

A vacuum system was set to pull an air flow of 1050 cc./min. by inserting the tapered end of a standard capillary tube through the dental
25 dam of the cigarette holder and adjusting the reading on the water manometer to the correct RTD. The water level of the manometer was set at zero before inserting the standard
30 capillary.

Then, the butt end of a cigarette or plug was inserted to a depth of 5 mm. in the dental dam of the cigarette holder. The pressure drop behind this cigarette with 1050
35 cc./min. of air flow passing through was read directly as RTD (inches water) from the inclined water manometer.

The molecular weight described in this specification is an average molecular weight
40 and is determined as follows:

(1) Preparation of phthalic anhydride-pyridine reagent: Add 42 g. of phthalic anhydride to 300 ml. of freshly distilled pyridine

(3) Calculations:

grams sample $\times$ 200

(B-A)N

=average molecular weight

90 A=ml. of N normal NaOH required for the sample

B=average ml. of N normal NaOH required for the control

WHAT WE CLAIM IS:—

95 1. A filter for tobacco smoke comprising synthetic filaments or fibres, activated carbon particles and a high molecular weight polyalkylene glycol (as hereinbefore defined).

2. A filter according to claim 1, comprising synthetic filaments which are at least in part
100 cellulose acetate.

3. A filter according to claim 2, wherein

contained in a one-quart brown glass-stoppered bottle. Shake the bottle vigorously until complete solution is effected. The reagent preferably should stand overnight before using; however, the solution may be heated under hot tap water until a slight cooling of the reagent occurs, indicating complete reaction. 45

(2) Procedure: Prepare a sufficient number of clean dry heat-resistant pressure bottles to make all control and sample determinations in duplicate. Carefully pipet 25 ml. of the phthalic anhydride-pyridine reagent into each
50 of the bottles, using the same pipet for each transfer. Reserve two of the bottles as blanks. 55

Into each of the other bottles introduce the specified amount of sample in the manner indicated. Make all weighings to the nearest
60 0.1 mg.

Fit the bottles with pressure stoppers and wrap each securely in a canvas bag. Place the samples and blanks as close together as possible in a water bath maintained at $98 \pm 2^\circ\text{C}$. for 30 minutes. Maintain sufficient water in the bath to just cover the liquid in the bottles. Remove the bottles from the bath and allow them to cool in air to room temperature. Do not remove the wrappers from the bottles while they are hot, or attempt to hasten the cooling by immersing them in cold water, as a serious accident can result from the breakage of the bottles. When the bottles have cooled, loosen the wrappers, uncap to release any pressure, and then remove the wrappers. 70

To each bottle add exactly 50 ml. of standard 0.5 N sodium hydroxide, using the same pipet for each addition. Be sure to allow the same drainage time for each bottle as this amount is not considered in the final calculation. Add 5 drops of a 1.0 per cent pyridine solution of phenolphthalein indicator and titrate with standard 0.5 N sodium hydroxide to a pink endpoint permanent for at least 15 seconds. 75



2000

the cellulose acetate filaments each have a denier of from 3 to 6.

4. A filter according to any preceding claim wherein the activated carbon is coconut carbon. 105

5. A filter according to any preceding claim wherein the activated carbon particles have a U.S. standard mesh size of from 10 to 44.

6. A filter according to claim 5, wherein the size of the activated carbon particles is such that 95% are retained on a 30 U.S. standard mesh screen and 95% pass through a 12 U.S. standard mesh screen. 110

7. A filter according to any preceding claim, wherein the high molecular weight poly- 115



alkylene glycol is polyethylene glycol, polypropylene glycol or methoxypolyethylene glycol.

5 8. A filter according to any preceding claim which contains from 2 to 200 parts by weight of carbon particles, and from 2 to 25 parts by weight of a high molecular weight polyalkylene glycol per 100 parts by weight of synthetic filaments or fibres.

10 9. A filter for tobacco smoke according to claim 1, substantially as herein described.

15 10. A filter for tobacco smoke constructed and arranged substantially as described herein with reference to and as illustrated in the accompanying drawings.

11. A smoking article comprising tobacco

and a filter as claimed in any one of claims 1-9.

12. A process for preparing a tobacco smoke filter as claimed in claim 1, which comprises 20 coating or partially coating synthetic filaments with a high molecular weight polyalkylene glycol (as hereinbefore defined), applying to the coated or partially coated synthetic filaments a coating of carbon particles, and 25 forming the filaments into the required shape within a wrapping material.

13. A process according to claim 12, substantially as hereinbefore described.

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14, South Square,
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Leamington Spa: Printed for Her Majesty's Stationery Office by the Courier Press.—1967.

Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.

