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Glucam™* P-20 Humectant

Glucam™ P-20 in Fragrance Applications

FRAGRANCE FIXATION

Fixation, or the increase in the lasting power of a fragrance in appropriate vehicles, is an important consideration and usually a highly desirable property of a perfume compound. Particularly in alcoholic extracts marketed primarily for their fragrance value, fixation without distortion is a major goal. Traditionally, fixation is achieved in several ways:

- Floral and botanical absolutes, concreted, resinoids
- Animal secretions and extracts
- Macrocylic musks
- Nitro musks
- Low volatility aromatic components

In addition to various degrees of fixative power, most of these materials exhibit another major property. They have their own distinct odors that constitute an integral part of the final fragrance. They cannot be used to impart fixative properties without affecting the final odor.

Glucam™ P-20 humectant, a unique raw material derived from glucose, serves to increase significantly the lasting power of many fragrance types without imparting an odor of its own. Glucam™ P-20 humectant is completely soluble in water and alcohol and is partially or completely soluble in many oils, including perfume oils. It can be premixed with the perfume oil or dissolved separately in the finished product system. Its cost is well below that of most perfume ingredients, including the least expensive fixatives.

THE RESEARCH PROGRAM

In a study designed to demonstrate the properties of Glucam™ humectants, increased fragrance duration when Glucam™ P-20 humectant was added to certain commercial colognes, toilet waters and bath splashes was observed. Typical florals, herbals, citruses and lavenders demonstrated the most effect. Oriental, heavy woodsy and very expensive fragrances showed the least effect. Initial observations were made organoleptically on perfume blotters and confirmed by an extensive series of consumer panel tests, both on blotters and on skin. The tests were expanded to include product categories beyond alcoholic extracts, such as dusting powders, bar soaps, creams, room deodorizers, etc. These studies are the subject of U.S. Patent #4,264, 478, “Polyol Fragrance Fixatives”, April 28, 1981, assigned to Amerchol Corporation. Copies are available upon request.

The study consisted of a representative group of individual aroma components. A reproducible protocol to measure fixation at various time intervals was developed. The hypothesis was that very low-boiling chemicals would evaporate so rapidly that no fixative would afford significant retention. High boilers would evaporate so slowly that no fixation was really necessary. In between lay a large number of intermediate boilers covering a broad area of odor types vital to perfumery.

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Approximately 50 aroma components were selected to cover the entire boiling range to challenge the hypothesis. They ranged in boiling point from 142C to 350C. The hydrocarbon roots included aliphatics, aromatics, alkylaryls, terpenes, sesquiterpenes, heterocyclics, etc. The functional groups included esters, aldehydes, acetals, ketones, alcohols, hydrocarbons, olefins, phenols, lactones, ethers, etc.

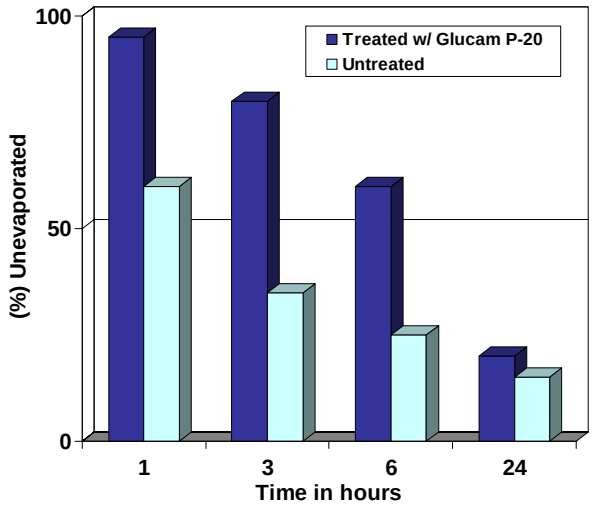
The experimental procedure entailed applying one milligram of the test material to a series of blotters and allowing them to evaporate for specified periods of time. The periods chosen were 1, 3, 6 and 24 hours. The blotters were extracted at the appropriate time with a suitable solvent and the amount of material remaining was determined by gas chromatography or ultra-violet spectroscopy.

RESEARCH RESULTS

The study established a clear pattern with some shading and overlap but no major anomalies. Boiling point was determined to be an important factor while chemical structure was not. The major point that emerged was that the perfumer would be able to get clear guidance in the design of his fragrance.

The research also clearly showed fixation by Glucam™ P-20 humectant. Typical data developed during the study is graphically illustrated below.

Fixative Effect on Eugenol



The data shows that Glucam™ P-20 humectant has a major fixative effect on many aroma components, including the following shown in ascending order of boiling point.

- Linalool
- Citronellal
- Decyl Aldehyde
- Benzyl Acetate
- Menthol
- α-Terpineol
- Linalyl Acetate
- Isobornyl Acetate
- Menthanyl Acetate
- Citral
- Geraniol
- Trans Anethole
- Linalyl Butyrate
- Safrole
- Caryophyllene
- Geranyl Acetate
- Hydroxycitronellal
- Indole
- Cinnamic Aldehyde
- Eugenol
- Diphenyl Oxide
- Methyl Anthranilate
- Cinnamic Alcohol
- β-Ionone
- Aldehyde C-16
- Yara Yara
- Amyl Salicylate

Very low and very high boiling aromatics conformed to predictions and were essentially unaffected by treatment with Glucam™ P-20 humectant. As the boiling point rose, degree of fixation shifted toward the 6 and 24 hour end of the time exposure scale, but it remained clearly evident throughout the broad middle of the boiling point range.

THE BENEFITS

The chief reason for using Glucam™ P-20 humectant is its proven fixation of fragrance. When used for this purpose, it will also provide significant added benefits that enhance the properties of fragranced products.

In addition to the benefits described below, Glucam™ P-20 humectant can be used in treating perfume grade alcohol to reduce the typical solvent pungency.

Economy and Cost Reduction

Glucam™ P-20 humectant is much less expensive than the traditional fixatives and it also provides major savings in total perfume costs. The incorporation of Glucam™ P-20 humectant in fragrance products and a variety of other products using fragrance permits the reduction of fragrance oil used by 5% to 20%. For example, 5% concentrations of perfume oil in a cologne can be reduced to 4.5% or even 4.0% with no loss in perceived intensity. Considering the very high percentage of ingredient cost due to the perfume oil, the potential savings are significant.

Emollient and Humectant Properties
As do most humectants, Glucam™ P-20 humectant attracts moisture but it exhibits an additional property that other humectants do not. It serves as an emollient (skin softener, moisturizer) either when used alone or in combination with other parts of an emollient system. Thus, traditional humectants such as glycerine, propylene glycol, sorbitol, etc. may be replaced by Glucam™ P-20 humectant which serves a multiple role.

Sting Reduction
The addition of Glucam™ P-20 humectant to alcoholic or hydroalcoholic extracts (e.g. aftershave, cologne, bath splash) normally applied over large, sensitive skin areas significantly reduces the usual stinging sensation.

Ease of Incorporation
Glucam™ P-20 humectant is readily soluble with simple stirring in alcohol and water in all proportions. It can be mixed with many perfume oils (except those rich in hydrocarbons). It is readily formulated into a wide variety of fragrances, cosmetics, toiletries and household products.

APPLICATIONS

Because of its special properties, Glucam™ P-20 humectant is strongly recommended in finished products whose primary function is fragrance or in any product containing fragrance where increased duration of fragrance or reduced concentration for cost savings is desired.

- perfumes
- toilet waters
- colognes
- aftershaves
- bath products
- shampoos
- skin care products
- bar and liquid soaps
- dusting powders
- room deodorizers
- sachets

THE PRODUCT

CTFA Name: PPG-20 Methyl Glucose Ether

Chemical Description: Twenty mole propxylate of methyl glucose

Physical Description: Pale, yellow, practically odorless, medium viscosity liquid

Glucam™ P-20 Humectant Fragrance Fixation Experimental Data

ABSTRACT

Quantitative evidence of fragrance fixation by a 20 mole propxylate of methyl glucoside (Glucam™ P-20 humectant) was obtained by subjecting a representative series of pure aroma chemicals to accurately measure evaporation and retention studies over time intervals of 1, 3, 6 and 24 hours.

EXPERIMENTAL PROCEDURE

Evaporation Procedure: Perfume blotters are spotted with a 1 mg of an aroma compound with and without 1 mg of the Glucam™ P-20 humectant. The methanolic solution of the aroma compound, with and without of the Glucam™ P-20 humectant, is applied to the top of these perfume blotters by means of a 10 ul syringe or micropipettor. These blotters are then placed in a "fish tank" where they are allowed to evaporate at ambient room temperature, pressure and humidity, for periods of 1 hour, 3 hours, 6 hours and 24 hours. At the end of each specified time period, the top portion of each blotter is cut off and placed into a suitable vial or flask containing methanol. The aroma compound is extracted from the blotter with methanol.

The resulting solution is then appropriately analyzed by G.L.C. or U.V.

G.L.C. Procedure: Each perfume blotter strip is extracted with 1.00 ml of methanol containing 1 mg of an "internal standard" for more reliable quantitation.

The amount of aroma compound in each blotter solution is determined by comparing the G.L.C. response ratio of aroma compound/internal standard in the blotter solution to a standard solution deliberately prepared to have a similar response ratio. A number (2 to 4) of G.L.C. injections of each blotter and standard solution are made and the results are averaged.

U.V. Procedure: Each perfume blotter strip is extracted with 2 to 100 ml of methanol depending upon the U.V. absorption of each individual aroma compound. The amount of aroma compound in each blotter solution is determined by comparing the U.V. absorbance of each blotter solution to a standard solution. The amount of aroma compound in each solution is proportional to the absorbance of each solution.

EXPLANATION OF DATA

The aroma chemicals in the attached table are listed in ascending order of boiling point.

While not all the boiling points are exact, for the purposes of this study, they serve as very useful guides. Where the symbol "±" is used, it implies that no boiling point was found in the literature but that the approximate boiling point listed positions it properly in the table. In actual practice, the great majority of the materials listed are distilled at reduced pressures since they tend to decompose at their boiling points.

After the boiling point column, there are four double columns under the overall heading of "Percentage Unevaporated." Each double column is headed by a time in hours, specifically 1, 3, 6 and 24. Within each time column there are two columns – one labeled "T" meaning that it has been treated with the fixative, and one labeled "U" meaning that nothing has been added to the aroma materials. The numbers in each column represent the percentage of material remaining on the blotter, subsequently extracted and measured for each aroma chemical in each time area. Wherever there is a dash, it means that no detectable amount of the material could be extracted and that it effectively is a zero.

The first entry in the table is n-Propyl Acetal with a boiling point of 123°C. After 1 hour with both treated and untreated materials, only 1% remained on the blotter. Beyond that, no material was detected. This material is so fleeting that nothing can serve to fix it significantly.

The last entry in the table is Benzyl Cinnamate with a boiling point of 350°C. In this case, all the readings – treated or untreated – 1 hour or 24 hours – are essentially 100%. This material is so nonvolatile that it does not require fixing. Just inside these two extremes are a number of materials which tend to show relatively little effect by the fixative. There is, however, a larger number of materials which show varying and frequently dramatic effects.

Citronellal has a relatively low boiling point of 206°C. However, 40% of it is retained after one hour of evaporation when treated with Glucam™

P-20, while only 17% of the untreated material remains. This implies a very significant effect on the Citronellal note, particularly in the early stages of its presence on the wearer's skin. After 3, 6 and 24 hours, both the treated and untreated show unevaporated residues of 10% or less, implying that most of the effect of the materials is gone after a couple of hours and fixation is no longer a question. But the effect in the first hour, which is the most significant period to the wearer, is dramatic.

Benzyl Acetate is a common workhorse ingredient in many floral fragrances, with a boiling point of 215°C. After 1 hour, the treated material retains 46% of its strength while the untreated material drops to 11%. After 3 hours, the Benzyl Acetate is essentially gone, but here too, its effectiveness in that critical period is demonstrated. Linalyl Acetate, another staple in many perfume compositions, retains 43% of the treated material after 1 hour while the untreated is down to 8%. It too is essentially gone after 3 hours. Similar results are noted for Isobornyl Acetate and Methanyl Acetate. As the boiling points continue to climb, the basic pattern persists but changes in total duration of fragrance begin to show.

Citral shows greater than 60% retention when treated and about 40% when untreated. This is a significant differential and means that good retention is obtained in that early critical stage of fragrance perception by the user. After 3 hours, there is no longer the complete disappearance of the fragrance component seen in lower boiling chemicals and there is a leveling off in the difference between treated and untreated. By the time the 6 hour level is reached, most of the material is gone.

For Geranyl Acetate, the difference between the treated and untreated after 1 hour is 8%, but after 3 hours the difference becomes more significant – 55% for the treated and 32% for the untreated. The same relationship continues at 44% compared to 20% for 6 hours and then levels off without complete loss after 24 hours to approximately 25% for both treated and untreated.

Hydroxycitronellal boils at 250°C and shows a smooth, steady relationship between the treated and untreated: significant after 1 hour, more pronounced after 3 and 6 hours, and still showing after 24 hours.

Methyl Nonyl Acetaldehyde has a boiling point of 252°C but demonstrated a very dramatic difference in behavior compared to other materials boiling in the same general range. After 1 hour, the treated material remains 54% unevaporated whereas the untreated material has lost all but 3%. After 3 hours the material is essentially gone. The result may appear to be anomalous, but it may also mean that there is more to the entire phenomenon than mere vapor pressure depression. While one could theorize all sorts of mechanisms such as acetal formation or other types of bonding, suffice to say that the empirical results show an unusual effect with this branched chain aliphatic aldehyde.

Eugenol and Diphenyl Oxide are in the same general boiling range as Hydroxycitronellal and Methyl Nonyl Acetaldehyde, but they tend to conform to the pattern shown by Hydroxy-citronellal. They very clearly demonstrate a quantitative fixative effect.

Yara Yara has a boiling point of 274°C and shows a very low loss on evaporation after the first hour. There is a modest effect at 1 hour and no significant effect after 3 hours but a fairly significant effect after 6 hours with almost complete loss after 24 hours.

Vetiverol, with an approximate boiling point of 285°C, shows that the material requires no added fixation for high retention. After 1 hour, treated or untreated, approximately 80% remains and even after 24 hours, the figures for both remain in the upper 60's. This effect becomes more pronounced with Aldehyde C-14 with the bulk of the material still there after 24 hours.

Ethyl Vanillin demonstrates very little volatility with losses of only 3 to 7% after 24 hours.

Returning to the table, materials such as the sesquiterpene alcohol, Santalol, show essentially no evaporation losses in the 24 hour period.

TABLE

AROMATIC CHEMICAL	Boiling Point °C @ 760 mm	PERCENTAGE UNEVAPORATED							
		1 Hour		3 Hours		6 Hours		24 Hours	
		T	U	T	U	T	U	T	U
n-Propyl Acetal	123	1	1	--	--	--	--	--	--
Isoamyl Acetate	142	4	5	--	--	--	--	--	--
Alpha Pinene	156	16	12	8	6	--	--	--	--
Benzaldehyde	180	20	23	20	22	18	20	15	15
Linalool	199	29	6	4	5	3	3	3	3
Acetophenone	202	8	6	<1	<1	--	--	--	--
Citronellal	206	40	17	9	9	7	10	5	6
n-Decyl Aldehyde	208	40	19	16	14	16	13	14	5
Allyl Caproate	±210	3	5	3	5	--	--	--	--
Benzyl Acetate	215	46	11	2	--	--	--	--	--
Menthol	216	45	25	16	10	4	5	2	3
Alpha Terpineol	217	58	38	19	11	5	8	--	--
Phenylethyl Alcohol	219	82	79	57	51	31	26	2	2
Linalyl Acetate	220	43	8	1	1	1	1	1	1
Methyl Salicylate	221	18	19	9	10	9	11	8	9
Isobornyl Acetate	225	40	17	2	2	<1	<1	--	--
Menthanyl Acetate	227	32	12	1	1	--	--	--	--
Citral	229	63	41	27	23	7	8	5	7
Geraniol	229	94	56	62	44	52	36	--	--
n-Decanol	231	55	67	38	37	40	23	3	5
Trans Anethole	233	50	25	10	8	7	10	6	8
Linalyl Butyrate	235	74	65	32	8	6	1	--	--
Safrole	236	64	36	16	<1	--	--	--	--
Caryophyllene	240	68	56	24	1	--	--	--	--
Geranyl Acetate	242	82	74	55	32	44	20	25	26
Hydroxycitronellal	250	72	57	53	36	33	9	6	2
Indole	252	75	51	55	36	45	20	19	3
Methyl Nonyl Acetaldehyde (Aldehyde C-12M)	252	54	3	4	1	--	--	--	--
Cinnamic Aldehyde	253	77	68	47	28	27	20	16	18
Eugenol	253	91	65	76	33	58	21	21	16

AROMATIC CHEMICAL	Boiling Point °C @ 760 mm	PERCENTAGE UNEVAPORATED							
		1 Hour		3 Hours		6 Hours		24 Hours	
		T	U	T	U	T	U	T	U
Diphenyl Oxide	255	68	61	38	10	13	3	1	1
Methyl Anthranilate	255	94	95	71	35	52	28	20	21
Cinnamic Alcohol	258	89	85	82	67	74	54	37	27
Beta Ionone	±260	98	95	79	69	55	35	4	1
Heliotropine	263	87	88	72	73	51	47	28	32
Aldehyde C-18 (Gamma Nonalactone)	263	88	87	67	62	47	32	9	10
Aldehyde C-16 (Ethyl Methyl Phenylglycidate)	271	97	95	94	88	94	65	51	29
Yara Yara (Beta Naphthyl Methyl Ether)	274	89	82	50	48	47	24	4	5
Amyl Salicylate	280	95	86	85	73	67	44	10	1
Vanillin	285	85	89	62	56	60	42	60	46
Vetiverol	±285	84	82	76	69	71	67	69	66
Amyl Cinnamic Aldehyde	290	102	98	93	90	89	80	54	32
Ethyl Vanillin	±295	101	100	98	102	94	100	97	93
Aldehyde C-14 (Gamma Undecalactone)	297	100	100	98	97	87	91	74	66
Santalol	301	109	110	108	110	110	106	101	86
Coumarin	302	107	104	95	85	89	90	64	55
Benzyl Benzoate	323	95	94	93	91	93	91	84	76
Aurantiol	>325	95	100	86	92	75	70	42	38
Benzyl Cinnamate	350	101	99	101	102	100	98	102	99