Polyquaternium-69: A New Fixative Polymer with Enhanced Styling Benefits

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Abstract

PVP and PVP/VA have long been the mainstay of traditional styling gels and mousses but the rapid release of new hair care launches and the drive for product differentiation on the shelves has created demand for expanded performance of styling resins. In order to achieve improved performance, a new polymer has been designed by polymerization of vinyl pryrrolidone, vinyl caprolactam, DMAPMA, and the alkylated quaternary DMAPMA+- C₁₂H₂₅CL-, INCI designation -Polyguaternium-69 (AguaStyle™ 300). Improvements have been achieved in humidity resistance, shine, frizz control and mechanical properties without compromising basic attributes such as gel clarity, stability, mousse foam properties, and low VOC requirements. Polyquaternium-69 is a hydrophobic polymer, yet water soluble and predominately non-ionic with a weakly cationic nature providing a broad range of compatibility with cosmetic ingredients including anionic gellants. Polyquaternium-69 can be easily incorporated into various styling products such as clear and cream gels, aerosol and non-aerosol mousses, styling lotions, and sprays.

Performance data on this polymer and formulations substantiate the efficacy of these various applications. Specifically, improved water resistance for strong hold has been demonstrated by high humidity curl retention. The enhanced mechanical properties have been characterized by texture analysis and supported by both subjective panel and Salon testing. Increased shine and anti-frizz effects are captured using digital image analysis.

Introduction

Competition is heating up in the hair care market. 1,322 products were launched globally in 2006 in the hair styling category alone. Styling trends range from 'Bed Head' to 'Wet

Looks' to 'Spiked Hair' to 'Natural Look'. Each product is launched for a particular target market that has been carefully studied by market researchers. In such a dynamic world, formulators increasingly face the challenge to create formulas that provide unique performance benefits in a cost-effective manner.

Polyvinylpyrrolidone (PVP) and copolymers of vinyl acetate or acrylates (VA) are some of the active ingredients commonly used in hair fixative and styling products to provide 'hold' or 'stiffness' to the hair. In addition, they provide formulation flexibility when used in combination with polyacrylate type gellants such as carbomer. However, consumers want more. Styling products need to provide all day hold and durability of hold after a mechanical action such as combing and maintain hair style under high humidity. To address this market demand, we have developed a new polymer by copolymerizing vinyl pyrrolidone and vinyl caprolactam with quaternized and alkylated moieties. This new polymer is Polyquaternium-69 (AquaStyle™ 300).

Study results reported in this article demonstrate that AquaStyle $^{\text{TM}}$ 300 provides enhanced styling benefits compared to traditional systems in styling products including clear and cream gels, aerosol and non-aerosol mousses, styling lotions and sprays.

Summary of key benefits in styling applications:

- · More durable and elastic hold
- · Increased shine and lustre
- Reduction of frizz
- Excellent high humidity curl retention
- Synergistic with hydrophobic gellants
- Extremely low flaking
- Broad raw material compatibility



Figure 1

Appearance	Yellow viscous liquid
Percent Solids	28.0-32.0
Percent Moisture	54-59
Percent Ethanol	12.0-17.0
Molecular Weight	~350,000

Chemistry

AquaStyle[™] 300 is a patented¹¹, water-soluble, quaternary polymer composed of vinyl caprolactam (VCL), vinyl pyrrolidone (VP), dimethylaminopropyl methacrylamide (DMAPMA), and methacryloylaminopropyl lauryldimonium chloride (MAPLDAC). The structure and typical properties are portrayed in Figure 1.

Each of the monomeric components of this polymer were specifically chosen and their relative properties optimized to provide the special attributes to AquaStyle[™] 300. Vinyl pyrrolidone provides initial stiffness on hair after drying. Vinyl caprolactam increases the hydrophobicity and provides increased film flexibility and durability of hold. DMAPMA imparts conditioning and provides smoothness of the film and flexibility of hold. And finally MAPLDAC, hydrophobically

modified quaternary monomer, provides durability, substantivity and the associative behaviour of the polymer with hydrophobically modified gellants. Together, these monomers combine to provide the enhanced styling benefits of AquaStyle™ 300 as will be shown through the experimental results below.

(MAPLDAC)

Rheology

Rheology is a key attribute of styling gels for consumers. Styling gels are pseudoplastic in nature to enable easy removal from the jar, ensure smooth application on the hair, and prevent 'dripping' from the hand.

Rheology and resulting gel properties were tested by combining AquaStyle™ 300 with a number of typical rheology modifiers. Most interesting was the study for high clarity gels, combining AquaStyle™ 300 with a hydrophobically modified gellant – Carbopol® Ultrez 21. Once dispersed in water the tightly coiled cross-linked polyacrylic acid opens up as the polymer hydrates, building viscosity by filling space with swollen micro-gels. Upon neutralization with a suitable base, the alkyl groups of both AquaStyle™ 300 and Carbopol® Ultrez



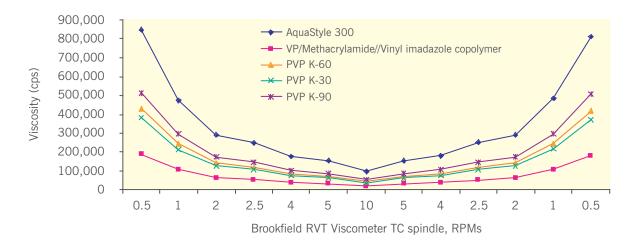


Figure 2: Comparison of Pseudoplastic Rheologies 4% Styling Polymer in 0.5% Carbopol® Ultrez-21

21 freely associate to form a matrix that creates a synergistic boost in viscosity. This is shown in Figure 2.

Synergy

A simple pure-mixture design experiment¹² was run to determine that a true viscosity synergy exists between AquaStyle[™] 300 and Carbopol[®] Ultrez 21. In a pure mixture design experiment, the response depends only on the relative proportions of the ingredients present in the mixture and not on the amount of the mixture. When the total amount is constant, then the behaviour of the measured response is purely a

function of the combined blending properties of the ingredients in the formula.

Figure 3 details the interaction of AquaStyle™ 300 with the hydrophobically modified gallant Carbopol® Ultrez 21 to build gel viscosity in a synergistic way. It should be noted homologues of PVP or the competitive benchmark VP/Methacrylamide/Vinyl Imadazole Copolymer do not deliver this added beneficial association when combined with hydrophobically modified gellants.

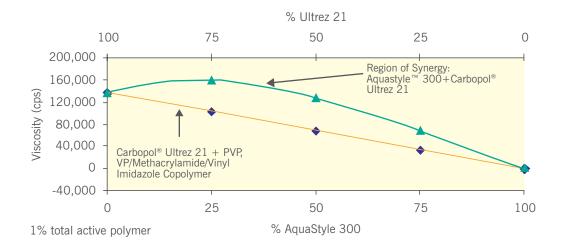


Figure 3: Viscosity Synergy between AquaStyle™ 300 and Carbopol® Ultrez 21



Performance on Hair

A combination of subjective and instrumental tests was conducted to determine the effectiveness of AquaStyle™ 300 compared to typical styling products including:

- High Humidity Curl Retention
- Hair Characteristics Test
- Flexibility and Durability
 - o Dynamic Hair Spray Analysis
 - o Cantilever 3-point Bending
- Water Resistance
 - o Tress Dunk test
- Hair Shine
 - o Image Analysis
- · Anti-frizz Effects
 - o Image Analysis
- Gel clarity

High Humidity Curl Retention

High humidity resistance is a necessary factor for hair styling products for creating and maintaining a hairstyle. A study was conducted to determine the humidity resistance of a hair gel containing 4% solids AquaStyle™ 300 versus a commercial gel containing a high load of PVP and VP/VA Copolymer.

The test protocol involves treating the curls with the styling polymer. The curls are then stored in a humidity chamber and

monitored at 80°F and 90% relative humidity. As shown in Figure 4, a dramatic difference is seen in the high humidity curl retention of AquaStyleTM 300 versus the commercial hair gel.

Curls treated with the commercial product start to fail after only one hour, whereas curls treated with the AquaStyle™ 300 gel have over 90% curl retention even after 24 hours.

Hair Characteristics Test

Hair Characteristics testing is conducted to determine stiffness and other important hair attributes of styling resins. In this test, a styling gel containing AquaStyle™ 300 is added in a controlled fashion to hair tresses and dried overnight. A trained panel then assesses a series of hair characteristics of the curls against set criteria using a scoring system of one to ten, where a score of one indicates a very soft, natural-like hair feel and a ten indicates very, very stiff hair feel.

As represented in Figure 5, a carbomer gel containing 4% solids AquaStyleTM 300 exhibits higher stiffness and stiffness durability than the commercial product containing PVP. Performance was comparable against the PVP, VP/VA Copolymer spiking gel. These styling properties are achieved with higher shine and less flaking when compared to the commercial product.

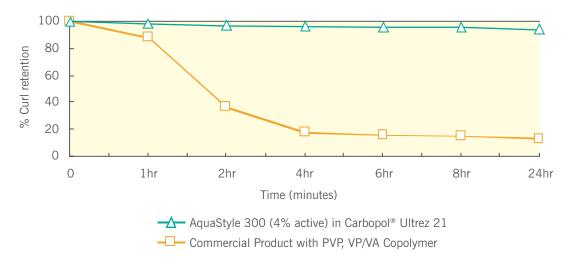


Figure 4: High Humidity Curl Retention after 24 Hours @ 90% RH, 80°F



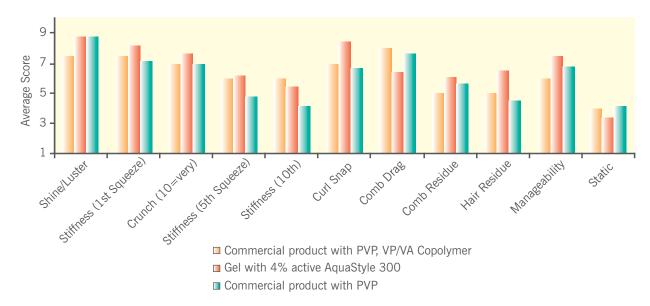


Figure 5: Hair Characteristics Score of "10" is preferred

Flexibility and Durability of Hair

Studies conducted using the Dynamic Hair Spray Analysis and Cantilever 3-Point Bending method determine the flexibility and durability of hair treated with styling polymers when subjected to mechanical force such as combing. The instrument used is a texture analyzer.

Dynamic hair spray Analysis

The method utilizes tresses formed in Omega loops. The method has been well documented by Jachowicz et al.¹⁻⁵. Briefly, hair is formed into omega loops. A set quantity of material is applied onto the hair by a pipette and allowed to dry under controlled humidity. The mechanical properties of the treated omega loops are then measured with the texture analyzer. The probe is programmed to depress the tress to a certain depth and then retract. As the probe goes through its cycle the texture analyzer measures the forces that the probe encounters. The following physical parameters are calculated from the force data:

 F_1 - maximum force in the 1st deformation

F₁₀ - maximum force in the 10th deformation

 F_{10}/F_1 - ratio of maximum forces in the 10th and 1st deformation

 E_{10}/E_1 - ratio of modulae in the 10th and 1st deformation

 H_{10}/H_1 - ratio indicating plasticity (scales from 1 (no plasticity) to 0 (doubling of the deformation))

Measurements are usually plotted as force vs. distance. A typical profile representing a brittle polymer is depicted in Figure 6a. It can be observed that the maximum force after one deformation of the probe on the hair results in an F_1 value of 335 grams and that breakage occurs before the probe is at its lowest point at 4 mm. The durability of the stiffness, F_{10}/F_1 , has a value of 0.66 which also indicates the brittleness of the polymer.

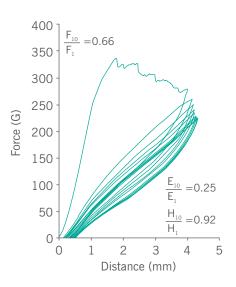


Figure 6a: Example of a Brittle Polymer



Polymers

The force vs. distance profile for the first cycle shows that there is little breakage of the polymer film on the hair as is evident for the brittle polymer. Also, F_{10}/F_1 is higher at 0.85 showing flexibility. This particular profile shows that the polymer is plastic, that is, there is a permanent deformation to the omega loop during the multiple cycles as indicated by the force starting at higher distances from the initial value.

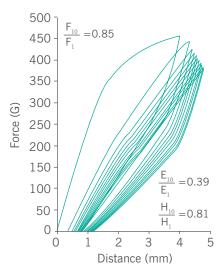
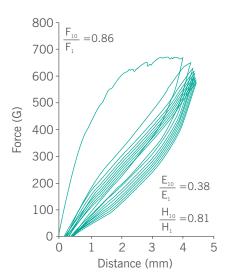


Figure 6b: Example of a Flexible and Plastic Polymer

4% active AquaStyleTM 300 was applied to the omega loop and dried under controlled conditions. When inspecting the shape of the force vs. distance profile it is evident that it is more similar to the model profile of a flexible polymer. There is little breakage of the polymer during the first cycle of depression of the probe of the texture analyzer. The initial stiffness after the first cycle, F_1 , has a high value at almost 700 grams, and its durability of stiffness or flexibility is high at 0.86. It is also seen to be slightly plastic in behaviour.

This instrumental method confirms the observations of the subjective panel tests which demonstrated that AquaStyle™ 300 has high initial stiffness as well as durability of hold after multiple compressions.



Aquastyle™ 300 (4% solids) Flexible and slightly plastic polymer

Figure 6c: Flexibility of Aquastyle™ 300 Polymer

Cantilever 3-point bending

In this method either a polymer solution or hair styling gel is applied to the hair tress and made into a ribbon with a special custom made clamping device. The hair is dried overnight at 40°C and then mounted on the apparatus. Again, as with the Omega loop test, the probe, in this case a triangular one, hits the tress on its downward cycle and depresses it a set distance. It is programmed to cycle up and down ten times to assess durability. As it goes through its cycle the texture analyzer measures the forces at work. The result is a profile of force versus distance for each of the ten cycles where the mechanical parameters can be calculated.

Two formulas were tested with the Cantilever Bending method: a simple gel containing 4% solids AquaStyle™ 300 thickened with Carbopol® Ultrez 21, and the same high solids PVP, VP/VA Copolymer commercial gel as tested in the HHCR and Omega Loop studies. Mechanical properties were assessed at both 50% and 90% relative humidity. Results are shown in Figure 7.

The results indicate that AquaStyle™ 300 is stiffer at 50% and 90% relative humidity conditions.



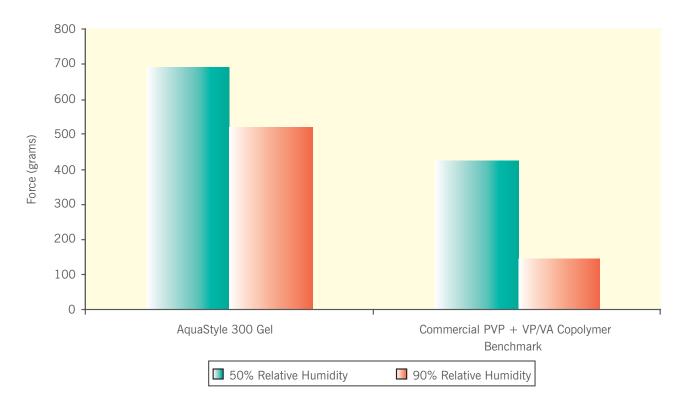


Figure 7: Cantilever 3-Point Bending Data F1 (max)

Measurement of other mechanical properties indicate that the AquaStyleTM 300 treated hair has significantly higher durability to stiffness (F_{10}/F_1), elasticity (E_{10}/E_1), and less plasticity (H_{10}/H_1) than PVP, VP/VA Copolymer treated hair.

This confirms the results obtained from the subjective panel tests (Hair Characteristics test) where panelists assessed the stiffness and crunch durability of curls treated with 4% AquaStyle™ 300 gel thickened with Carbopol® Ultrez 21 and 4% PVP K-90 gel. Results are shown in Figure 8.

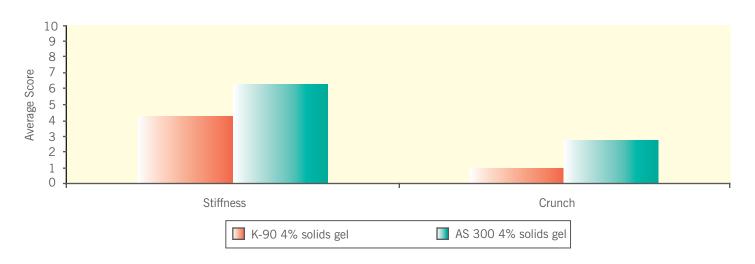


Figure 8: Stiffness and Crunch Durability to High Humidity 1 = Least, 10 = Most



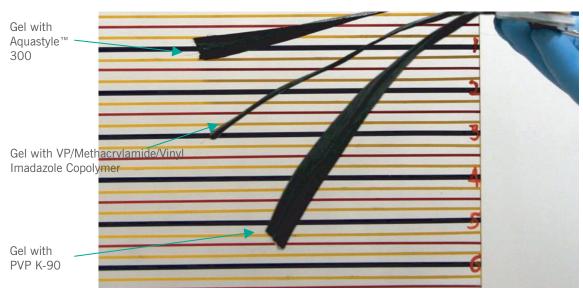


Figure 9

Water Resistance

Tress Dunk Test

Figure 9 highlights the hydrophobic effect of AquaStyle™ 300 on hair via the Tress Dunk test. The procedure consists of adding a polymer solution or hair gel to the hair tress, dried as a flat ribbon spike, submerged in water for 5 seconds and then held horizontally to demonstrate resistance to water.

Three hair gel systems were tested: PVP K-90, VP/Methacrylamide/Vinyl Imidazole Copolymer and AquaStyle™ 300. 0.5 grams of each gel was applied to a 3.5 gram tress and the experiment conducted. The rate of droop is measured against a calibrated board.

Figure 9 proves that hair treated with AquaStyle[™] 300 has the least droop and most water resistance. This is due to the hydrophobicity of AquaStyle[™] 300 versus the more hydrophilic and hygroscopic PVP and VP/Methacrylamide/Vinyl Imadazole Copolymer.

Hair Shine

Both visual observations of digital pictures as well as image analysis indicate that hair treated with AquaStyle™ 300 positively affects shine of hair. The details of the method are described fully by McMullen and Jachowicz⁶⁹. The method consists of situating a tress on a hemispherical barrel and

taking a digital picture of the hair surface with a high quality digital camera. Using image analysis software, the light intensity of the digital picture is scanned parallel to the fibre axis to produce a light scattering curve. Shine and lustre are then assessed by viewing the digital picture and the profile of the resulting scattering curve.

Figure 10 shows the digital pictures taken of two tresses, one is an untreated control and the other is treated with a 1% solids aqueous solution of AquaStyle $^{\text{TM}}$ 300.

It has been noted in previous work by McMullen and Jachowicz that when the reflection band appears more narrow, there is an increase in specular vs. diffuse reflection. This is evident in the AquaStyleTM 300 treated hair on the right, where it can be observed that the light and dark bands are more in contrast to each other as opposed to the untreated hair.

Image analysis on shine

Light intensity curves produced from image analysis of these digital pictures are portrayed in Figure 11. The peak intensities of the two curves indicate that there is a significant difference in lustre. The maximum specular reflection intensity for the polymer treated tress is 226 \pm 3, whereas the untreated control is 210 \pm 4. Previous research indicates that vinyl caprolactam contributes to the shine enhancement in styling products 6 .







untreated treated

Figure 10: Hair Shine

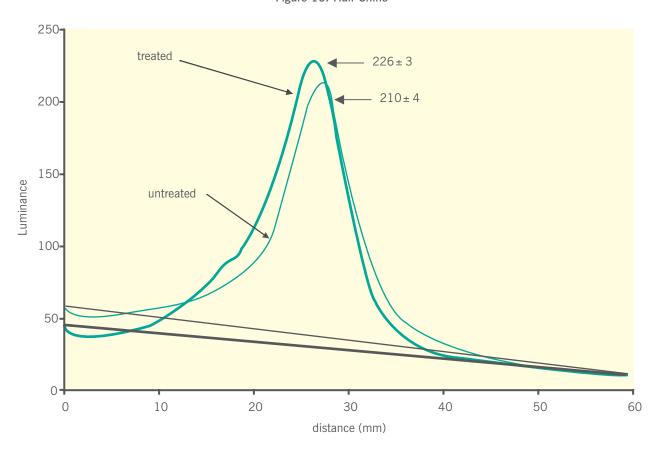


Figure 11: Image analysis on shine



Anti-Frizz Effects

Hair subject to high humidity tends to get frizzy. This is due to breakage of hydrogen bonds present between the protein chains that are responsible for keeping the hair in a particular configuration. However, when hair is coated with a hydrophobic resin layer such as AquaStyleTM 300, there is an increased resistance to moisture absorption resulting in less frizziness.

An experiment was conducted to test this effect. Dry, frizzy hair was treated with 0.3 grams:

- a) of a gel containing 4% solids AquaStyle™ 300 thickened using Carbopol® Ultrez 21
- b) PVP based commercial gel
 An untreated tress served as the control.

Time: Initial



Figure 12a: Untreated control

Time: After 8 hours



Figure 12b: Untreated control

All 3 hair tresses were put into a humidity chamber kept at 80°F and 90% relative humidity. Digital pictures were taken at 0 and 8 hours to capture the anti-frizz effect of the polymeric films.

As seen below, the untreated hair (Figure 12b) becomes frizzy due to absorption of water. On the other hand, hair treated with AquaStyle $^{\text{TM}}$ 300 (Figure 13b) counteracts this effect. This provides evidence that the hydrophobicity of this polymer provides a water resistant film which retards the negative effect of water absorptivity. Figure 14b shows that hair treated with the PVP-based commercial gel also exhibits a certain degree of frizziness after 8 hours.



Figure 13a: Hair treated with AquaStyle $^{\text{TM}}$ 300



Figure 13b: Hair treated with AquaStyle $^{\text{TM}}$ 300



Figure 14a: Hair treated with PVP-based commercial gel



Figure 14b: Hair treated with PVP-based commercial gel



Gel Clarity

Crystal clear gels with clarity above 85% transmittance can be formulated as shown in Figure 15 using 4% AquaStyle™ 300 and 0.35% Carbopol® Ultrez 21. Carbopol® Ultrez 21, also hydrophobically modified interacts positively with AquaStyle™ 300 to produce clear gels.



Figure 15

The processing of AquaStyle[™] 300 into gels is quite easy since it is flowable. However, dilution with water tends to make incorporation into the neutralized gellant easier. The AquaStyle[™] 300 has no neutralizable groups so the only alkali or amine necessary for the formulation is to neutralize the gellant. Typically, the gellant is fully neutralized and made uniform prior to the addition of AquaStyle[™] 300.

Formulating with AquaStyle ™ 300

AquaStyle[™] 300 can be readily incorporated into various styling products including clear and cream gels, aerosol and non-aerosol mousses, styling lotions and sprays. Since AquaStyle[™] 300 is only slightly cationic, it is compatible with a wide variety of nonionic, amphoteric and cationic polymers. Two prototype formulations – clear gel and aerosol mousse that were Salon tested are described overleaf.

Formula 1 is a clear hair gel based on 4% solids AquaStyle™ 300 which was the active level used as a basis for many of the

performance tests contained above. Glycerin acts as a plasticizer without negatively impacting styling performance as confirmed by the Cantilever 3-Point Bending method. This formula was also Salon tested vs. a well known commercial product with a high level of PVP, VP/VA Copolymer; the same as used in the subjective panel tests and instrumental analysis. Half head evaluations shows that our prototype with AquaStyleTM 300 increases shine, curl definition, conditioned feel, and style hold. Addition of glycerin also significantly reduces flaking as compared to the commercial product.

Formula 2 demonstrates AquaStyle[™] 300 usage in a mousse application. Here AquaStyle[™] 300 is used in combination with another styling polymer, Copolymer 845. Besides the primary styling benefits of the AquaStyle[™] 300, Copolymer 845 adds to the conditioned feel of hair both in the wet and dry state. Varisoft PATC is added primarily to raise the cloud point of the concentrate to enhance stability, but its auxiliary benefits are evident in increasing the quality of the foam and conditioning aspects to hair, both in the wet and dry state. The formula is made to be 6% VOC. Salon testing of this prototype was performed against a leading brand based on Polyquaternium-11. All wet and dry hair attributes were the same except that the AquaStyle[™] 300 mousse had increased curl formation and style memory.

Conclusion

AquaStyle™ 300 provides enhanced styling benefits when compared to other styling resins currently available without compromising basic formulation attributes such as clarity, stability, foam properties and utility in meeting low VOC requirements. The hydrophilic-hydrophobic balance in the polymer leads to excellent high humidity curl retention, increased durability of hold and anti-frizz effects under high humidity. The water-soluble nature of the polymer provides broad range compatibility with cosmetic ingredients including anionic gellants. As demonstrated through the different experimental studies, the multi-functional features of AquaStyle™ 300 provide enhanced styling benefits when compared to results obtained using traditional polymers and serve to address customer and market needs.



Formula 1 – Ultra Hold Hair Gel with Frizz Control With Aquastyle™ 300 #11275-62-1	
Ingredients	%W/W
Phase A	
Water	70.30
Glycerin	3.00
Acrylates/C10-30 Alkyl Acrylates Crosspolymer	
(Carbopol [®] Ultrez 21)	0.35
Aminomethyl Propanol (AMP-95)	0.30
Phase B	
Water	6.60
Polyquaternium-69 (Aquastyle™ 300)	13.40
Phase C	
Water	5.00
Disodium EDTA (Versene Na2)	0.05
PEG-40 Hydrogenated Castor Oil (Cremaphor RH-40)	0.50
Propylene Glycol (and) Diazolidinyl Urea (and) lodopropynyl	
Butylcarbamate (Liquid Germall® PLUS)	0.50
	100.00%

Procedure

- 1. Phase A Add water to main container and mix with moderate sweep agitation. Add Glycerin and mix until dissolved.
- 2. Disperse Carbopol $^{\circledR}$ Ultrez 21 and mix until uniform.
- 3. Add AMP-95 and mix until clear and uniform. Slow agitation when solution gets thick so as to not entrap air.
- 4. Phase B Premix AguaStyle™ 300 and water and mix until uniform.
- 5. Add Phase B to Phase A and mix until uniform.
- 6. Phase C Dissolve Na2 EDTA in water and mix until uniform. Add Cremaphor RH-40 and heat gently to about 40°C. Mix until uniform.
- 7. Add Phase C to main container and mix until uniform.
- 8. Add Liquid Germall® Plus and mix until uniform.

Physical Properties

Appearance Clear gel pH 7.45

Viscosity 42,000 cps (Brookfield DVII+, RVT, sp T-B @ 5rpm, 1 min., RT)

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Formula 2: – Aerosol Mousse with Aquastyle™ 300 and Copolymer 845 #11312-136	
Ingredients	%W/W
Phase A	
Water	78.35
Polyquaternium-69 (Aquastyle™ 300)	7.50
VP/Dimethylaminoethylmethacrylate Copolymer (Coplymer 845)	5.00
Palmitamidopropyltrimonium Chloride (Varisoft PATC)	0.65
Propylene Glycol (and) Diazolidinyl Urea	0.50
(and) lodopropynyl Butylcarbamate (Liquid Germall® Plus)	
Propellant	
Isobutane	4.88
Hydrofluorocarbon 152A (Dymel 152a)	3.12
	100.00%

Procedure

- 1. Add ingredients in order listed, mixing well between additions.
- 2. Fill in to cans, vacuum crimp and charge propellant.

Physical Properties

Appearance Clear liquid (form two separate phases when propellant added)

pH 6.59

Viscosity ~ 25 cps (Brookfield Spindle #2 LVT/12rpm/1min, 25°C)

Packaging

Aluminum Microflex or Phenolic lined cans, 02-15 Series - Mousse Spout, S90 0.024" stem, butyl gasket, inverted w/tailpiece 4 slot, and cut gasket Aluminum valve from precision.

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