Chapter V

Results and Discussions

This research work is aimed to determine the effect of mixing sequence in incorporating a builder into liquid detergent composition, effect of impeller types in the preparation of homogeneous liquid detergent, effect of mixing temperature on viscosity of liquid detergent composition, effect of mixing tank system and to determine the liquid detergent characteristics.

5.1 Effect of Mixing Sequence

Experiments were performed using standard tank configuration. Materials used in the formula are shown in Table 4.1. Materials having solubility limitation in liquid detergent composition were sodium carboxy methyl cellulose (CMC) and sodium tripolyphosphate (STPP). CMC when solubilized in water cause lumps of CMC-particles. [18]. Therefore, the dissolvation sequence of CMC was important and must be considered in this experiment. The other material that effect the homogeneity of the mixture was STPP builder, therefore the corporation of the builder into the liquid detergent composition had to be performed very carefully. The results of four types of mixing sequence effect were found to be as follow.

5.1.1 Sequence of Mixing Type 1

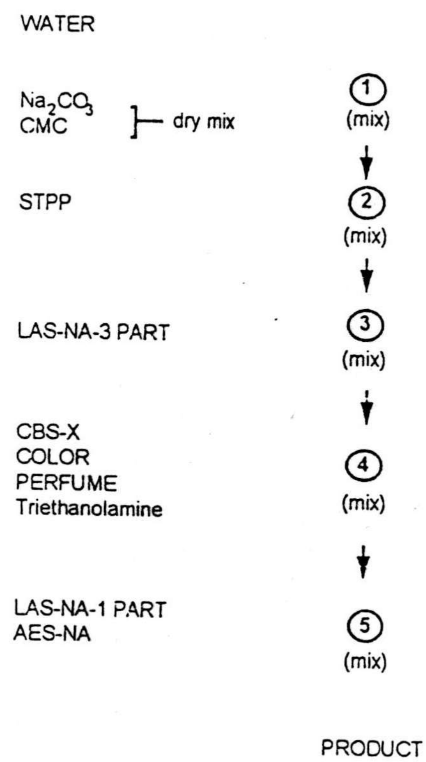


Figure 5.1 Sequence of Mixing Type 1

The liquid composition was prepared by feeding water to a vessel provided with a stirrer. The appropriate amounts of dry-mixed sodium carbonate and sodium carboxymethyl cellulose were introduced and mixed into the water with moderate stirring. In this step, gelling of CMC was obtained and are dispersed in the liquid. After that, sodium tripolyphosphate (STPP) was introduced into the vessel and mixed at higher stirring rate. As a result, STPP was hydrated and lumps of large solid particles were formed. These solid particles gathered near the baffles at the tank bottom and could not well be suspended. Thereafter the other materials were added. The final composition was not a homogeneous liquid.

5.1.2 Sequence of Mixing Type 2

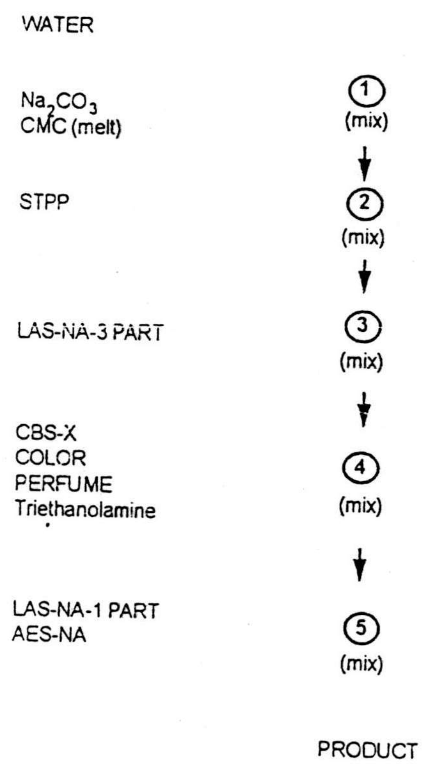


Figure 5.2 Sequence of Mixing Type 2

Purified water was charged into a vessel provided with a stirrer. Appropriate amounts of sodium carbonate were introduced and mixed into the water with moderate stirring until completely soluble. Melted form of CMC at 80 °C were added into the aqueous solution. Gel particles of CMC were obtained. After that, the other materials were added to the solution in the same sequence as the mixiate amount of sodium he dispersion of STPP was the same and liquid detergent composition was not homogeneous.

5.1.3 Sequence of Mixing Type 3

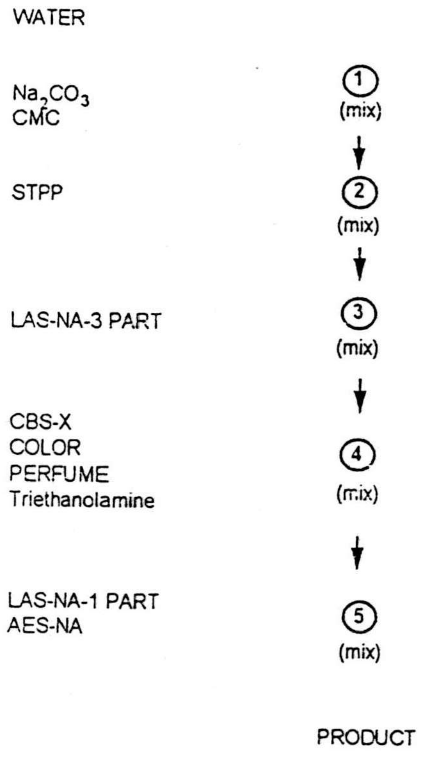


Figure 5.3 Sequence of Mixing Type 3

In the sequence shown in Figure 5.3, in the first step, sodium carbonate was completely dissloved in purified water, and then CMC powder was added and mixed into the aqueous solution with moderate stirring . CMC dispersed well and a slightly turbid solution was obtained. Thereafter, STPP was mixed into the aqueous solution with higher stirring and the results were the same as that obtained with mixing sequence type 1 and 2. After that all other materials were added. Liquid detergent composition was not homogeneous, some large particles of STPP still formed. This is because of the position of the impeller in the tank, if the impeller position is lowered as discussed in 5.2.1 stirring would be more effective. The product had a viscosity of 4900 cp. , a pH of 10.8 and the color and odor were stable after two months under ambient temperature and also at severe conditions. The sample which were stored in transparent plastic bottles at 20-25°C and ambient temperature, showed no phase separation. Only under severe condition, that is ,exposed in sunlight and in the oven at 45°C , slight separation was observed. When subjected to a freeze-thaw stability test, the composition remained stable.

5.1.4 Sequence of Mixing Type 4

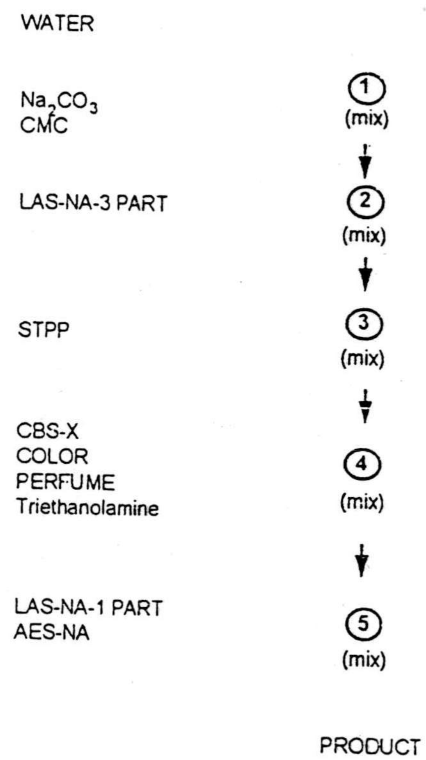


Figure 5.4 Sequence of Mixing Type 4

The first and the second step of sequence of mixing type 3 were repeated and aqueous solution of sodium carbonate and CMC was obtained. Thereafter 3/4 part of linear alkyl benzene sulfoanate (LAS) was added to increase liquid medium level and mixed with moderate stirring until a homogeneous solution was obtained. Then STPP was added and mixed into the liquid composition. Particles of STPP formed lumps of large solid particles and was not as well-mixed as the dispersion of sequence of mixing type 3. Thereafter the other materials were added. A white opaque, non-homogeneous liquid detergent was obtained having a viscosity of 2700 cp. and pH of 10.9 . Stability test results were similar to the liquid composition of sequence of mixing type 3.

5.1.5 Discussion on Effect of Mixing Sequence

The sequence of mixing for the preparation of liquid detergent built formula as reported in Thai Patent Application No.1134 [2] is the same as the sequence of mixing type 3 of this work. Dry-mixing of CMC and sodium carbonate or melt of CMC before adding into the water did not improve the solubility of this material . The important step of this sequence was the step of addition of sodium tripolyphosphate into the solution. Therefore, sodium tripolyphosphate should be added directly to the solution with continuous stirring. To avoid lump formation only fine particles of sodium tripolyphosphate should be used.

Not only the mixing sequence is important, but also the proportions of the materials used in the formula . In the Thai patent application [2] , the compostion should have a viscosity in the range of 2000 - 5000 cp. The composition of this experiment using standard tank configuration had a viscosity in the range of 2000 - 6000 cp. Consequently, viscosity of the composition could be decreased by using a hydrotrope material, such as sodium xylene sulfonate or sodium toluene sulfonate in the formulation as shown in US. Patent No. 4,228,043 [13] .

5.2 Effect of Impeller

The four impeller types used were turbine, paddle, propeller and 3-blade paddle and were compared for effects on mixing system for sequence of mixing type 3.

5.2.1 Turbine Impeller

As mentioned earlier in 5.1.3, the dispersion of STPP was not complete and it was due to the position of the stirrer in the standard tank configuration used.The configuration used was the standard tank configuration and the position of the stirrer in the tank was at the height of 1/3 of liquid height from the tank bottom. However, past researchers [10, 11, 12] showed that in order to disperse fine particles in a viscous fluid the stirrer should be closer to the bottom of the tank so that it will have more power to churn the fluid and disperse the particles more uniformly. Therefore, the height of the stirrer was reset to 1/5 of liquid height from the tank bottom. As a result of the adjustment after addition of STPP a homogeneous composition was obtained and had a viscosity of 3000 cp. Then 3/4 part of LAS was added to the composition and the viscosity decreased to 1000 cp. After that appropriate amounts of other additive ingredients such as, whitening agent, color, perfume and triethanolamine were successively introduced and mixed into the composition. The viscosity of the liquid composition after addition of additive dropped further to about 400-500 cp. Finally, the rest of LAS and appropriate amount of sodium alkylpolyethoxy sulfate (AES) were mixed into the mass. The composition became very viscous having a viscosity of about 2000-6000 cp.

In low viscosity liquid the disc-turbine radial impeller can pump more in the radial direction and separate the current in two portions, the upper and the lower portion. Therefore, the disc-turbine caused more discharging flow rate to achieve complete homogeneous mixing. But in high viscosity liquid, the disc-turbine could not homogenize well even when using high speed this is because the flow is not fully turbulent. One reason is that a change in flow pattern occurred when materials are mixed, the flow being confined to regions near the impeller [19]. To improve the mixing in order to obtain a well mixed composition two 6-bladed disc turbines were used. The distance between the two disc turbines was equal to the height of the first impeller from the bottom of the tank. As a result of this modification all liquid composition mixtures were homogeneous after continuous stirring.

The composition obtained was an opaque liquid having a viscosity of 2725 cp. , pH of 10.1, the color and odor was found to be stable for several months at various conditions. The composition stored in transparent plastic bottles at 20-25°C and ambient temperature (25-30 °C) remained stable, no separation being observed after 2 months. Except in severe conditions, exposed to sunlight and in an oven at 45°C, slight separation of the phases occurred. When subjected to a freeze-thaw stability test, the composition remained stable.

5.2.2 Propeller Impeller

The mixing time for the dissolution of water-soluble solid, sodium carbonate, using propeller type impeller was longer than the mixing time of other impeller at the same speed. It could be explained that impeller having same diameter and at the same speed, the propeller draw lesser power than most other impellers did, hence have less stirring efficiency. Therefore, for propellers higher speed had to be used in order to achieve a given horsepower and a particular pumping capacity . With regards to the dispersion of solid in this experiment, it was found that propeller was not a suitable impeller to use compared to other types of impellers.

5.2.3 Multiple Blade Paddle Impeller

Multiple blade paddle impeller was used in this experiment, homogeneous liquid composition was obtained until step 4 of the mixing seqence. In step 5 of the sequence LAS and AES were added and upon stirring the liquid compositon had high foam volume and higher liquid level than when using other types of impeller. Multiple blade paddles have a tendency tc increase liquid circulation therefore foam volume of the composition increased with stirring. In general, multiple paddles are suitable for high viscosity liquid mixing but in the preparation of this particular liquid detergent, which foams easily because of the surfactant, multiple paddle was not suitable.

5.2.4 Straight Blade Paddle Impeller

The mixing time for the dissolution of sodium carbonate in water using straight blade paddle impeller was shortest compared to other impellers types. This system could disperse STPP particles very well and no particles were found to adhere to the baffles in the tank. It could be concluded that impellers generating radial flow (such as disc-turbine , paddle) could achieve more complete mixing than impellers which generate axial flow (such as propeller). Same as all other experiments when LAS and AES are mixed into the liquid in the last step, the viscosity of product increased, this type of impeller, as with other types of impellers, was not able to mix the composition very well. Therefore, in the last step of the sequence, to obtain homogeneous liquid composition mixing was performed munually. The final liquid product had a viscosity of 6850 and a pH of 10.8. The composition remained stable the same as the composition of previous experiments.

5.3 Effect of Temperature

Temperature effect on viscosity of liquid composition at each mixing step was studied . Liquid composition was prepared by dissolving sodium carbonate and CMC in the purified water. Then sodium tripolyphosphate was introduced and mixed with high speed stirring. Temperature of water-bath was slowly adjusted. The viscosity of sample at 40 °C, 50 °C, 60 °C and 70 °C were measured. The results of this experiment are shown in Figure 5.14. The viscosity of sample increased slowly when temperature of system increased.

After the addition of 3/4 part of LAS and mixed into the composition, the viscosity of the sample decreased sharply from about 4000 to 1000 cp. this was because of an increase in water content of the aniònic surfactant. The effect of the temperature in this step was in contrast to the viscosity of the sample when STPP was added, as shown in Figure 5.14 . Viscosity of sample decreased when temperature of system increased. High temperature promotes good bonding between the surfactant and water, thus dissolution of the surfactant increased and led to low viscosity of the liquid composition.

After addition of appropriate amounts of additive ingredients, such as whitening agent, color, perfume and triethanolamine, were introduced and mixed until a homogeneous liquid mass was obtained. In this step, viscosity of liquid composition dropped to 400-500 cp. In the final step the rest of LAS and appropriate amount of AES were added and mixed into the composition, the viscosity of final liquid composition increased to 2000 - 6000 cp. The results of viscosity affect on the sequence of mixing are shown in Figure 5.15. Properties of liquid composition prepared using sequence of mixing type 3 at mixing temperature 60°C are shown in Table 5.7 and 5.8.

5.4 Effect of Mixing Tank System

Upon addition of LAS and AES in the final step all liquid compositions prepared had high viscosity and to obtain homogeneous composition in this step manual stirring had to be performed. Although the standard turbine and paddle type impellers could be used to homogenize the composition in the other steps but the final step could not achieved. It was because both types of impellers gave insufficient flow in high-viscosity mixture. For comparison, a T.K. AGI HOMO MIXER, which is a high speed heavy duty mixer having combination of stationary paddles and rotational paddles was used. The T.K. HOMO MIXER could completely mixed the ingredients and a homogeneous liquid composition was obtained. The paddle section is a U-shaped steel bar similar to the anchor type agitator which is usually used to mix high viscosity liquid.. The impeller provided more extensive flow and close-clearance design which gave a higher degree of wall scraping to eliminate the build up of unmixed materials at the wall and provided region of high shear for dispersing aggregates and lumps.

The properties and other characteristics of liquid composition depended on the rotational speed of the impeller. Normally the rotational speed of this type of impeller is quite low but the power required is high . At lower rotational speed (200 rpm), it was found that the mixing time for dispersing STPP was longer than the mixing time at higher rotational speed. Liquid composition was not a completely homogeneous mixture. At higher rotational speed (400 rpm), high foam volume of liquid mixture was obtained after the addition of surfactant and liquid composition became a high-viscosity mixture. At moderate rotational speed (320 rpm), mixing of the liquid composition was more complete than at lower speed.

Comparison of viscosity stability of liquid composition at various rotational speed of mixing are shown in the Figures 5.14, 5.17, and 5.20. All liquid compositions remained stable, same as the composition prepared in standard tank configuration.

5.5 Liquid Detergents Characteristic

5.5.1 Properties Comparison with Liquid Detergent in the Market

Heavy Duty Liquid Detergent Built Formula in the present market were first introduced by Fab Liquid Detergent of Colgate Palmolive (Thailand) Co.,Ltd. Second and third launched products were Breeze Ultra Liquid of Lever Brother (Thailand) Co., Ltd. and Biotex of Giwi (Thailand) Co.,Ltd. The properties of the liquid detergent composition of this experiment were compared to heavy duty liquid detergent built formula available in the Thai market, and are tabulated in Table 5.13. It can be seen that liquid detergent composition of this experiment has nearly the same properties as the Fab Liquid Detergent and Biotex. As for the Breeze Ultra Liquid, it has higher content of surfactant therefore the recommend dosage per time of washing was lower than other liquid detergent brand.

5.5.2 Comparison of Performance with Liquid Detergent in the Market

Data in Table 5.14 indicates the performance of several heavy duty liquid detergent built formula available in the Thai market . Performance test method performed was inaccordance to test methods of Thailand Instituted Standard (TIS 78-2528) [20] . Results showed similar washing power (detergency) of liquid detergent samples. Foaming properties are also similar. Effect of whitening agent in liquid detergent composition was similar, the cotton fabric when treated, became whiter and brighter than reference cotton fabric (without whitening agent coating on the fabric).

Table 5.1 Characteristic of Liquid Detergent Composition from the Sequence of Mixing Type 3

Typical Properties	Results
Active Matter (%)	15.1
Specific Gravity (30 °C)	1.098
Viscosity 30 °C (cp)	4900
pH (direct)	10.8

Table 5.2 Stability Test after 2 M of Liquid Detergent Composition from the Sequence of Mixing Type 3

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	4670	-	-	4920
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

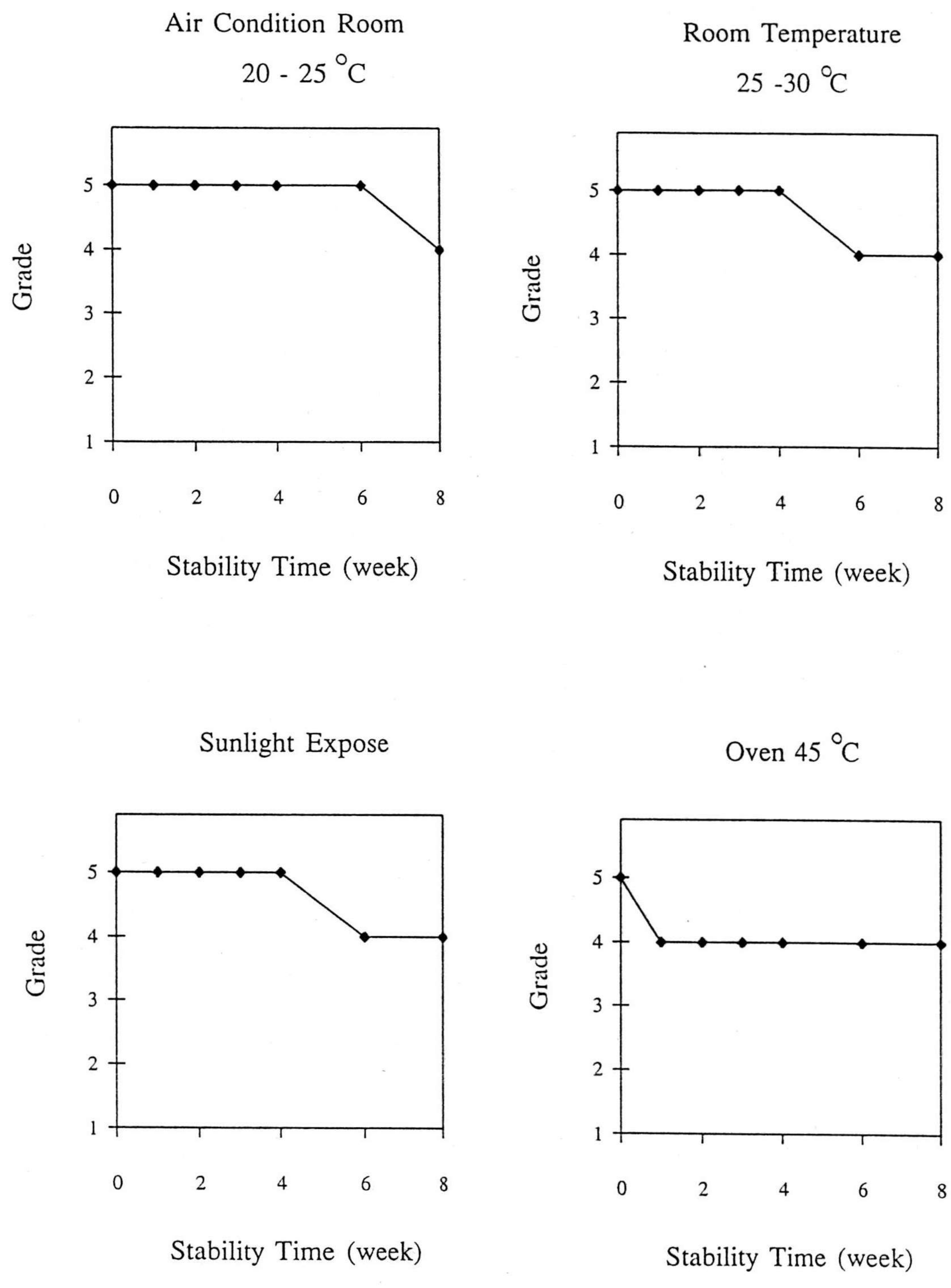


Figure 5.5 Odor Stability of Liquid Detergent Composition from the Sequence of Mixing Type 3

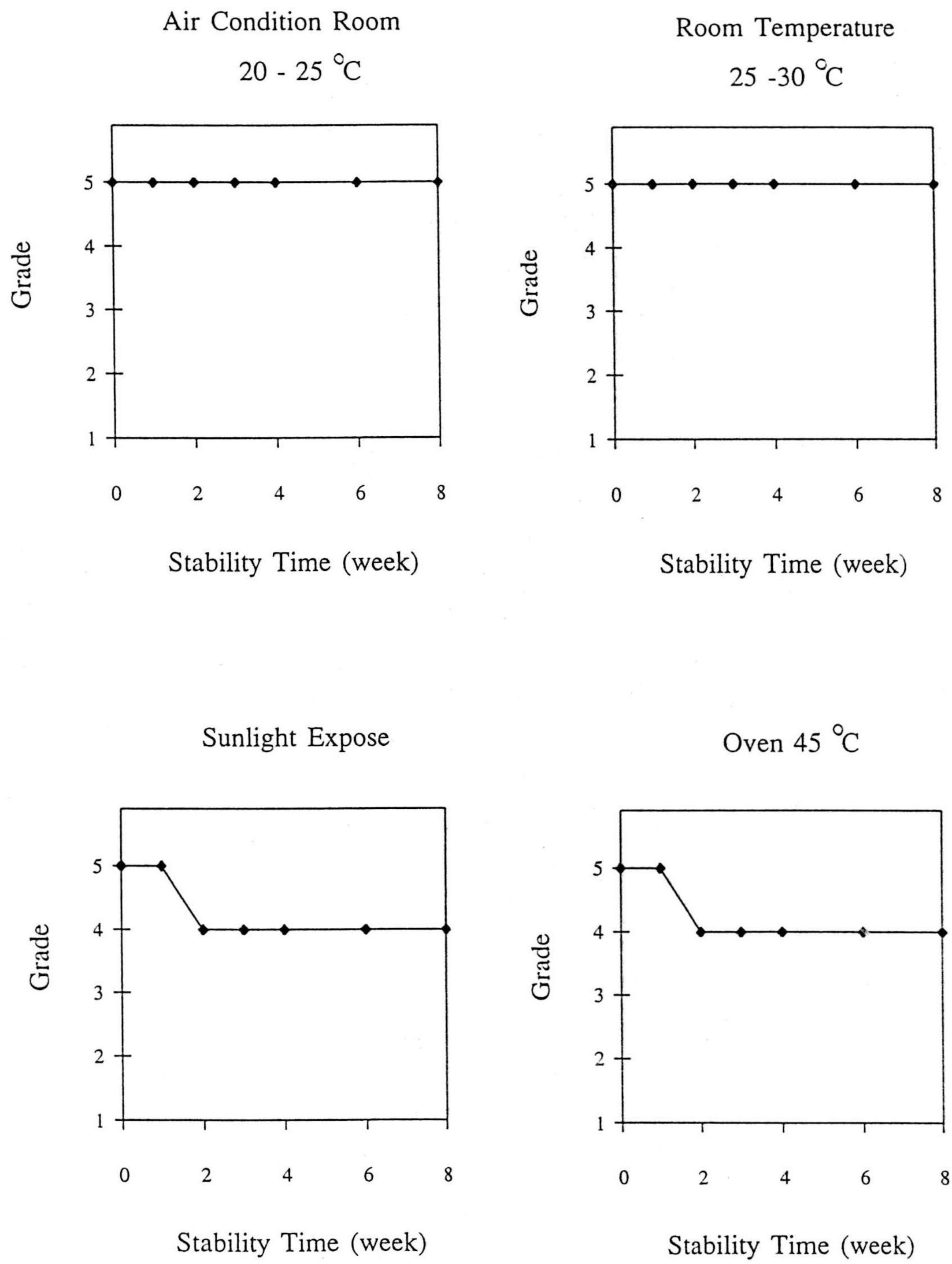
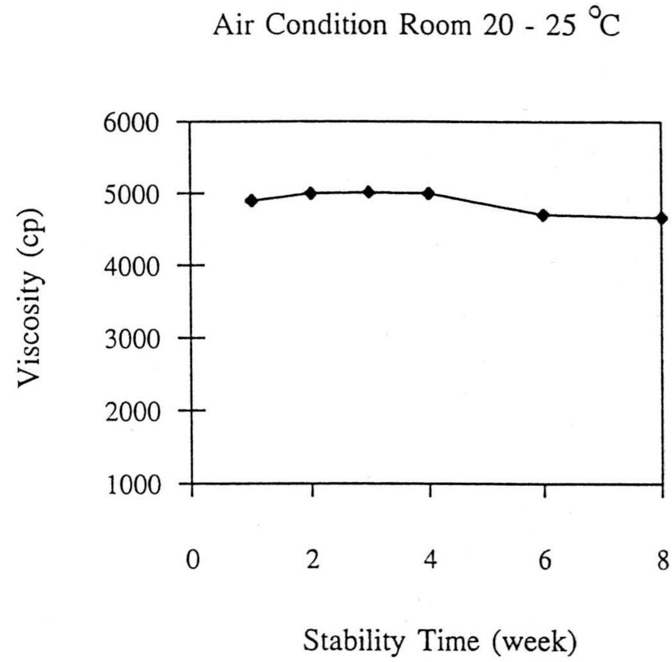


Figure 5.6 Color Stability of Liquid Detergent Composition from the Sequence of Mixing Type 3



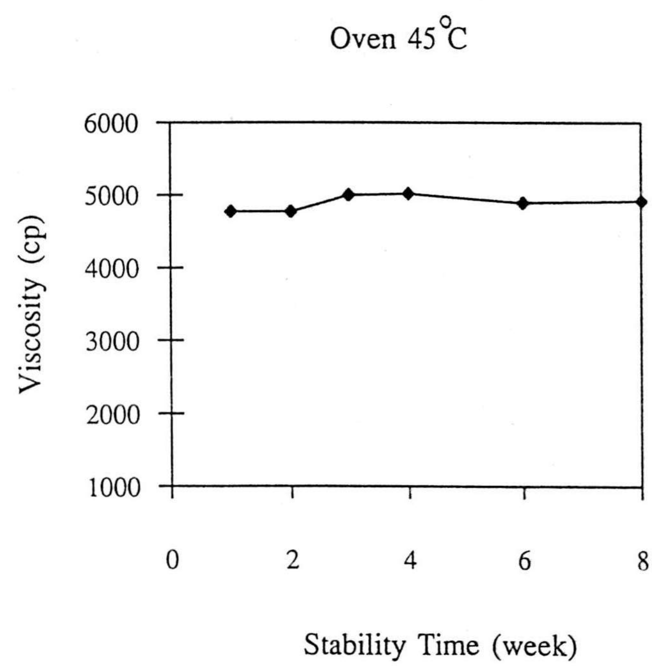


Figure 5.7 Viscosity Stability of Liquid Detergent Composition from the Sequence of Mixing Type 3

Table 5.3 Characteristic of Liquid Detergent Composition from the Sequence of Mixing Type 4

Typical Properties	Results
Active Matter (%)	14.8
Specific Gravity (30 °C)	0.9953
Viscosity 30 °C (cp)	2700
pH (direct)	10.9

Table 5.4 Stability Test after 2 M of Liquid Detergent Composition From the Sequence of Mixing Type 4

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	2400	2580	2520	3000
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

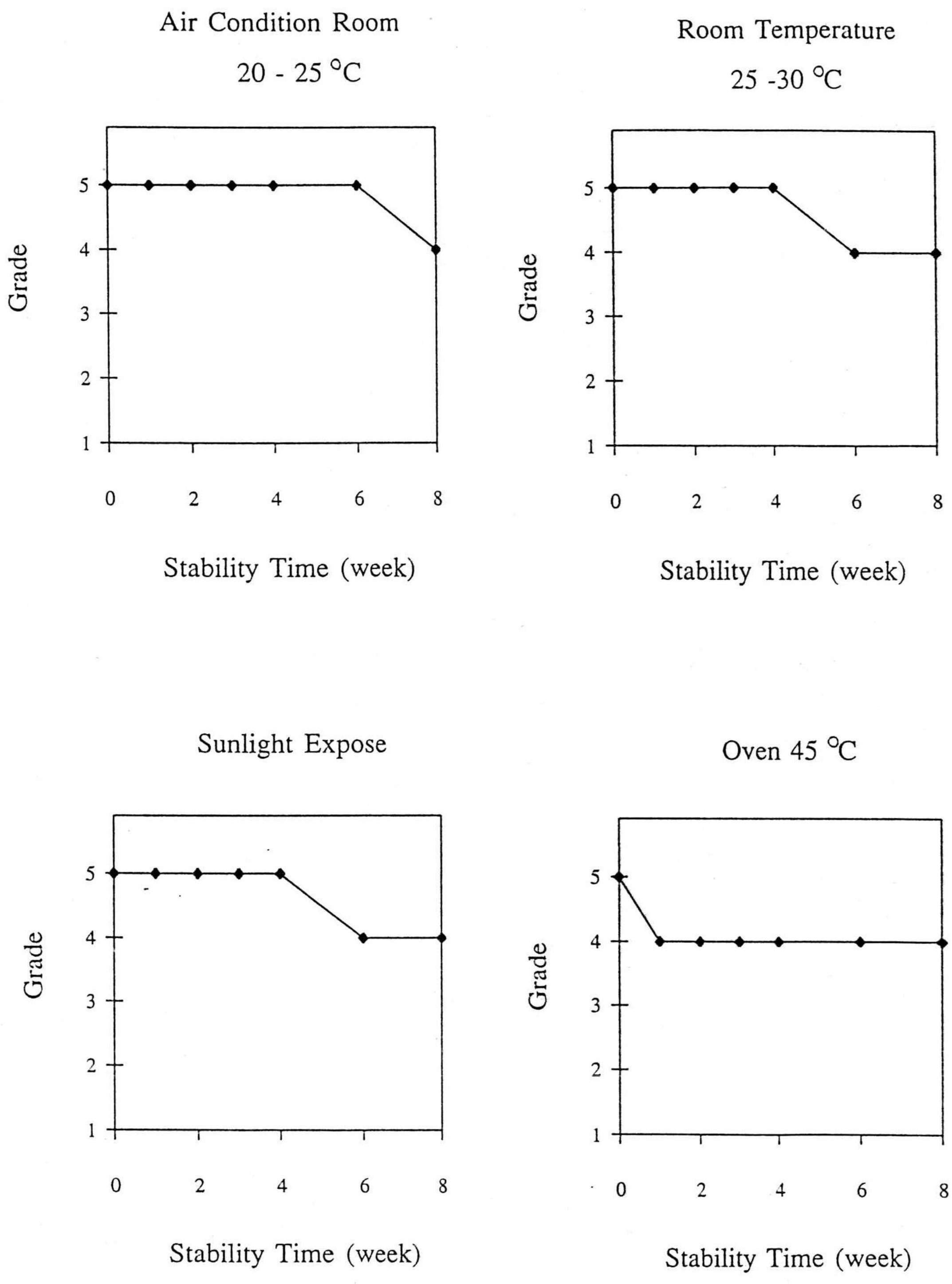


Figure 5.8 Odor Stability of Liquid Detergent Composition from the Sequence of Mixing Type 4

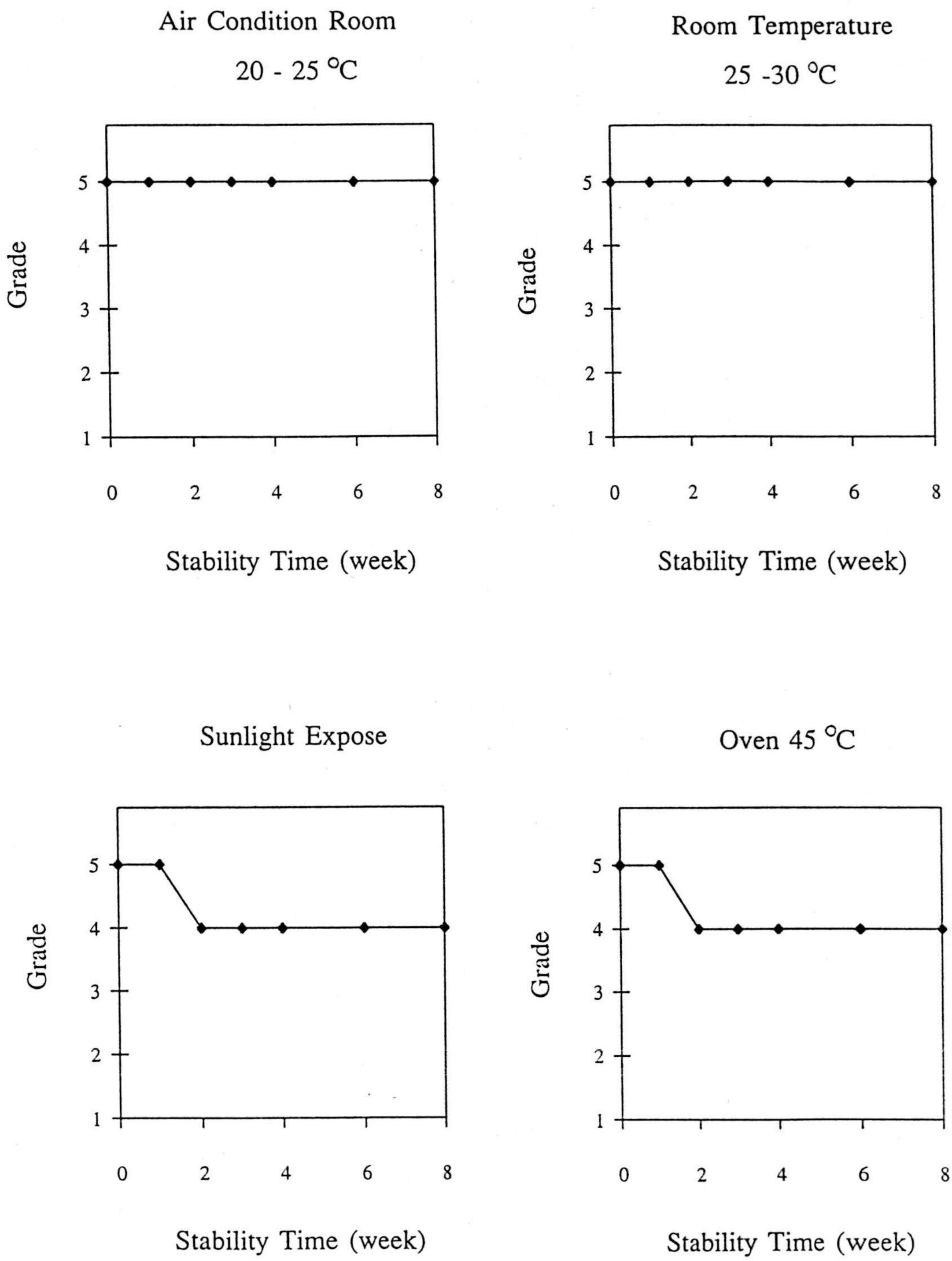


Figure 5.9 Color Stability of Liquid Detergent Composition from the Sequence of Mixing Type 4

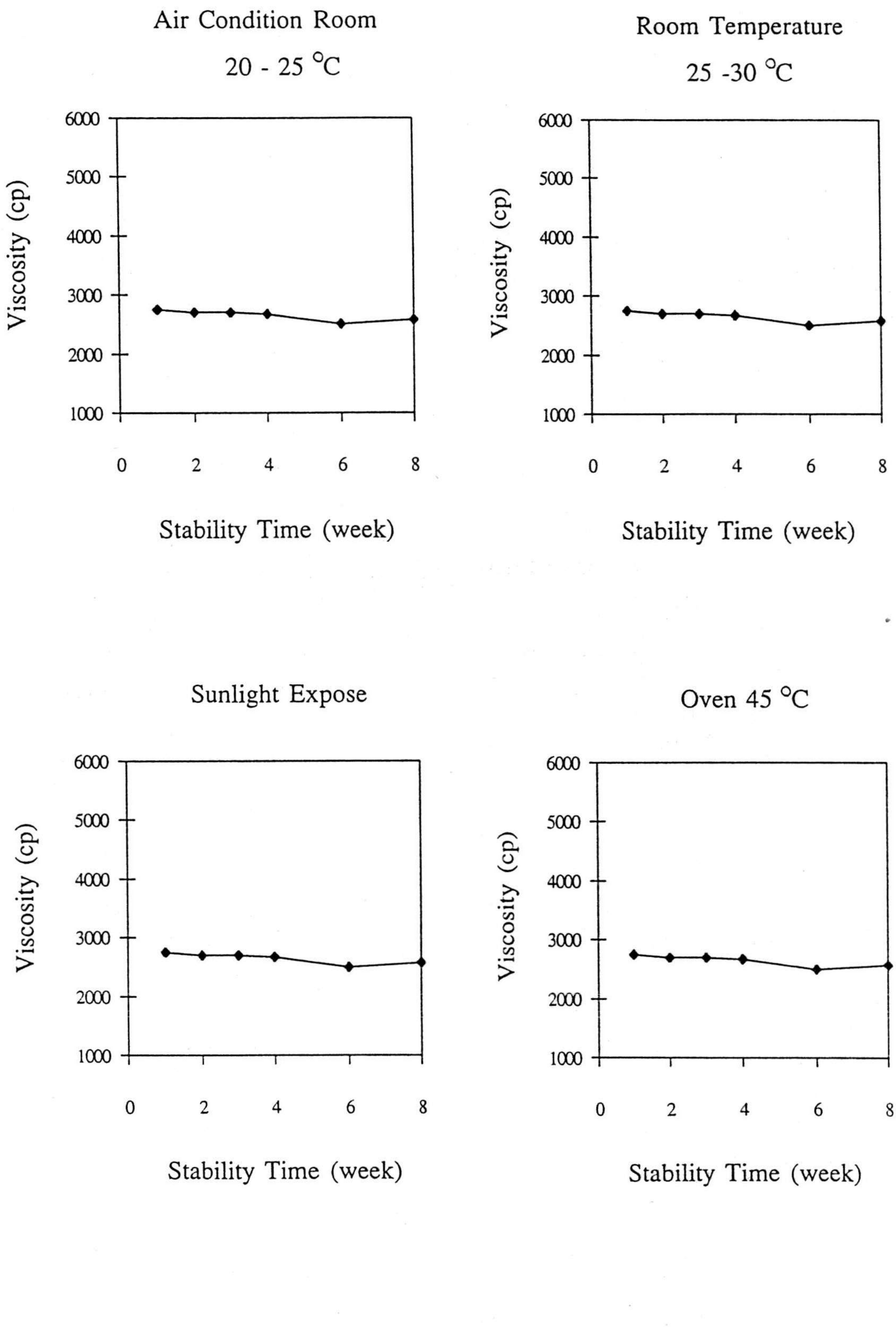


Figure 5.10 Viscosity Stability of Liquid Detergent Composition from the Sequence of Mixing Type 4

Table 5.5 Characteristic of Liquid Detergent Composition from the Turbine Effect on 5.2.1

Typical Properties	Results
Active Matter (%)	14.5
Specific Gravity (30 °C)	1.2058
Viscosity 30 °C (cp)	2725
pH (direct)	10.8

Table 5.6 Stability Test after 2 M of Liquid Detergent Composition From the Turbine Effect on 5.2.1

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	2300	2520	2400	2345
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

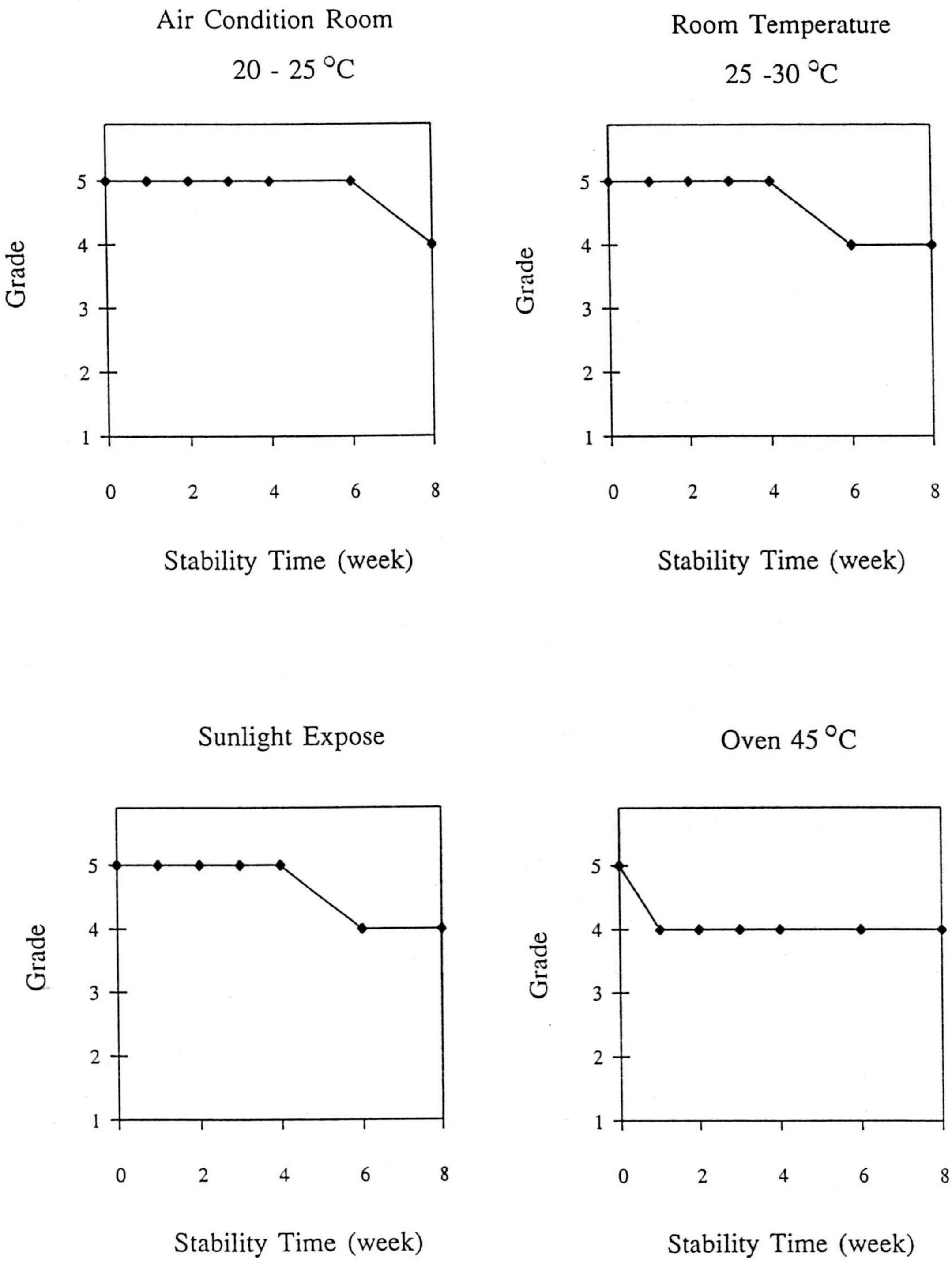


Figure 5.11 Odor Stability of Liquid Detergent Composition from the Turbine Effect on 5.2.1

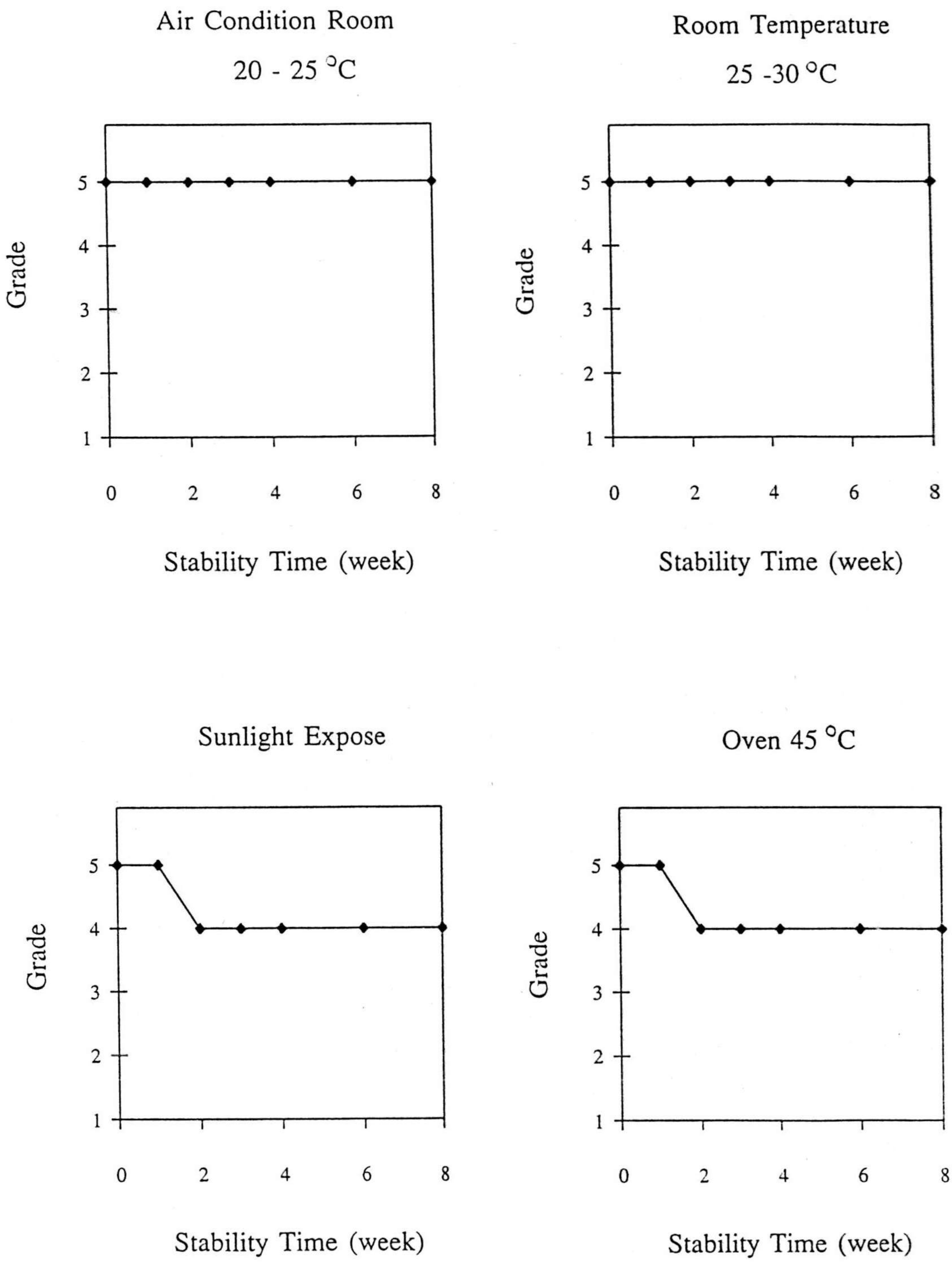


Figure 5.12 Color Stability of Liquid Detergent Composition from the Turbine Effect on 5.2.1

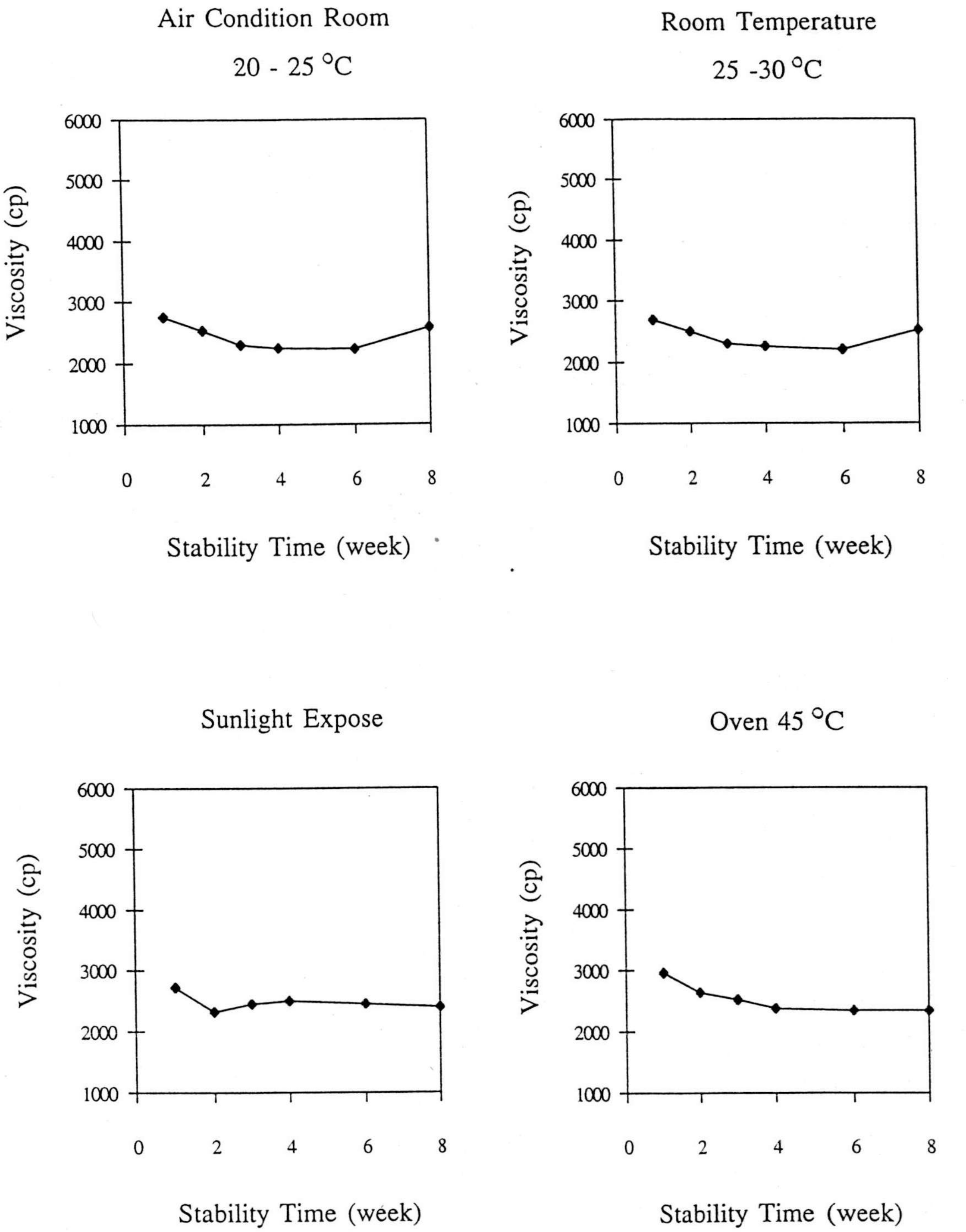
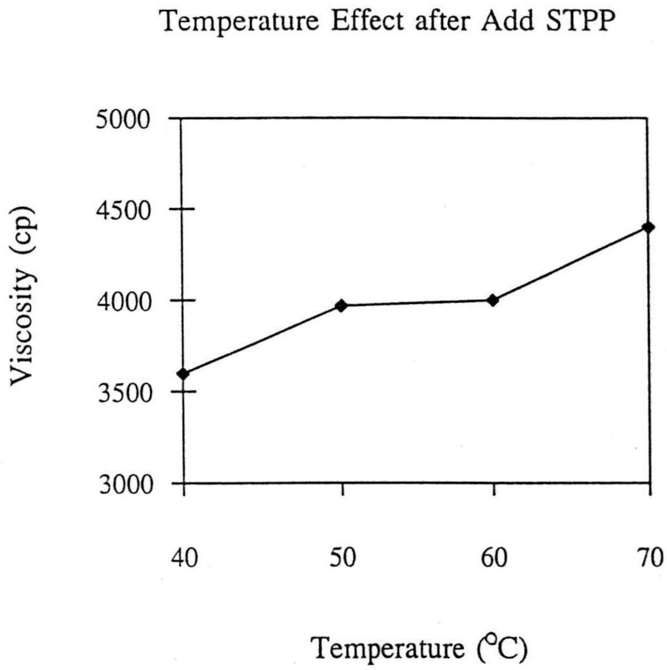


Figure 5.13 Viscosity Stability of Liquid Detergent Composition from the Turbine Effect on 5.2.1



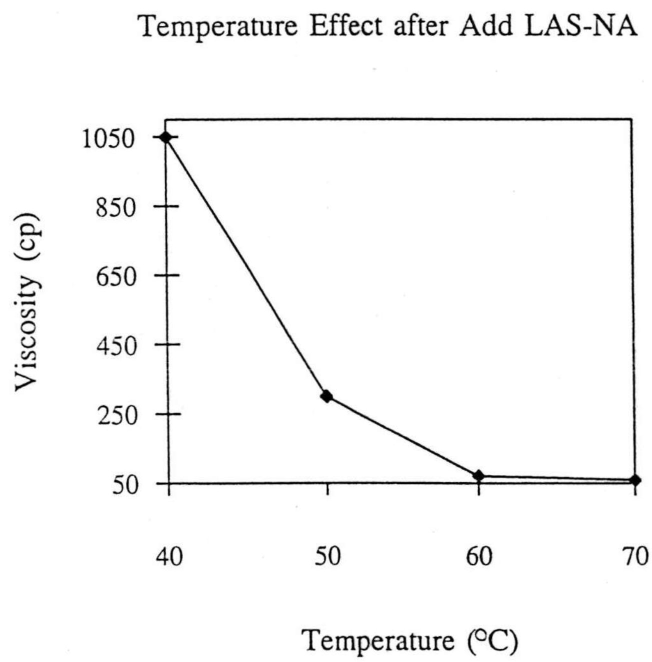
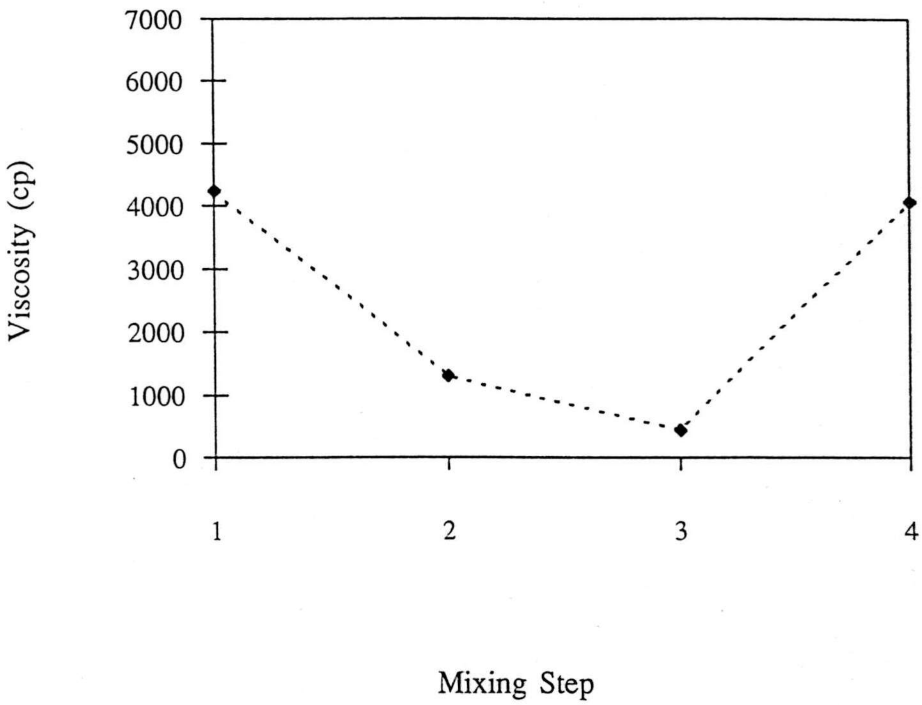


Figure 5.14 Effect of Temperature on Viscosity in Mixing Sequence



Step 1	Solubilize Sodium Carbonate
	CMC
	STPP
Step 2	After Add LAS-NA
Step 3	After Add Additive Ingredients
Step 4	After Add LAS-NA and AES-NA

Figure 5.15 Effect of Viscosity at Each Step of Mixing Sequence

Table 5.7 Characteristic of Liquid Detergent Composition at Mixing Temperature 60 °C

Typical Properties	Results
Active Matter (%)	14.8
Specific Gravity (30 °C)	1.1274
Viscosity 30 °C (cp)	5150
pH (direct)	10.5

Table 5.8 Stability Test after 2 M of Liquid Detergent Composition at Mixing Temperature 60 °C

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 4	5 -> 3
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	5490	5110	3405	7100
Separation	No Separation	No Separation	Slight Separation	Slight Separation
Freeze Thaw Test	NoSeparation	No Separation	No Separation	Noseparation

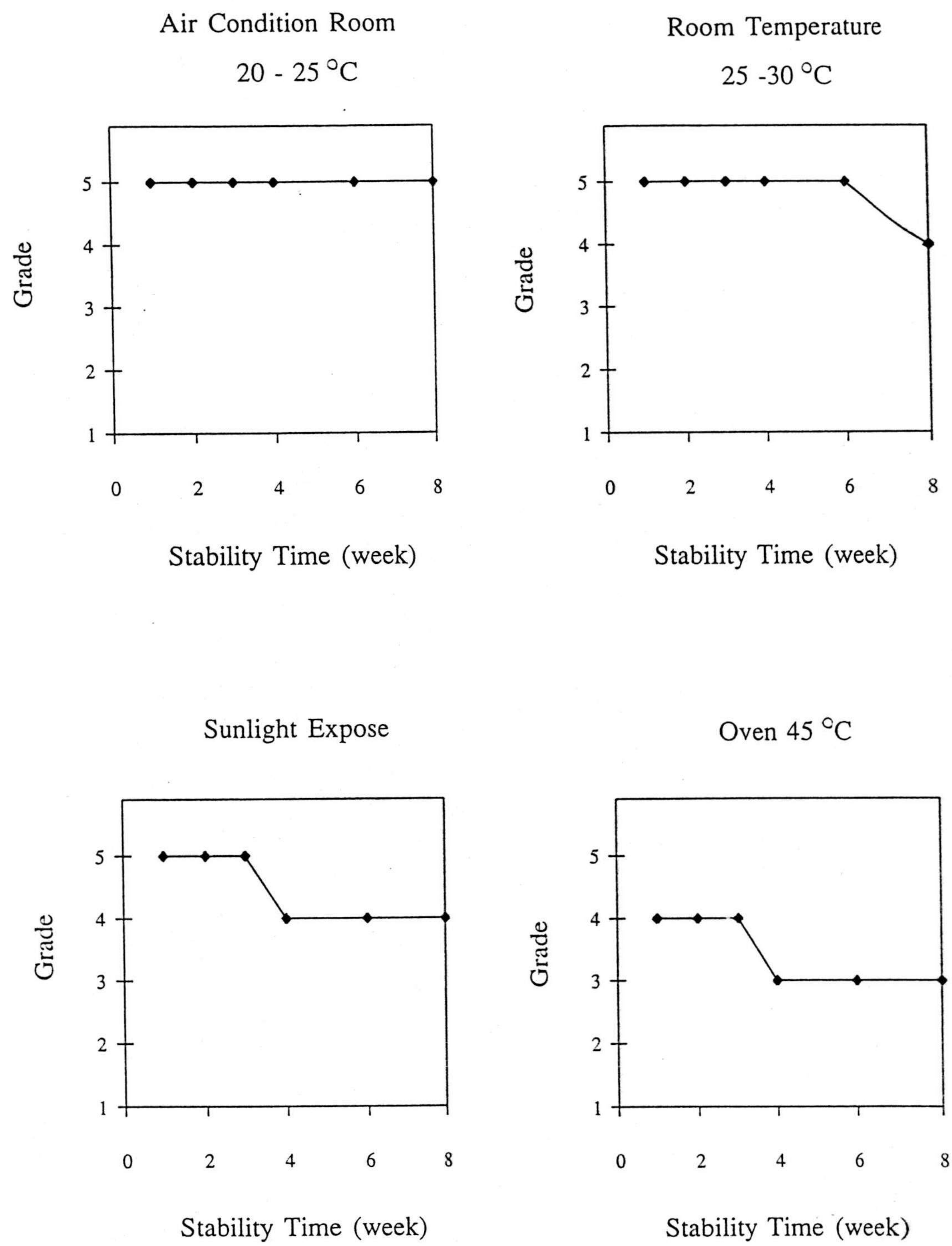


Figure 5.16 Odor Stability of Liquid Detergent Composition at Mixing Temperature 60 °C

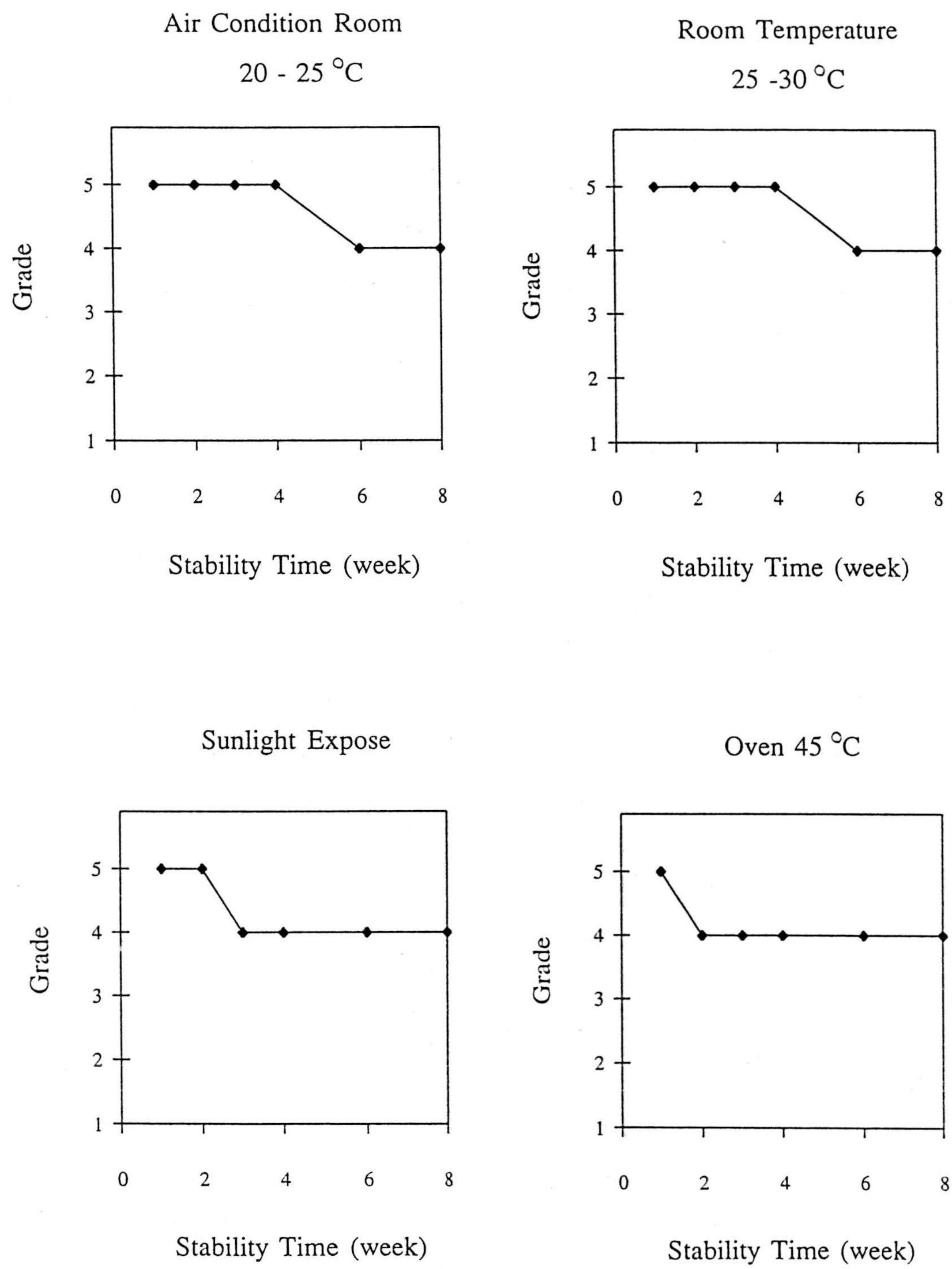


Figure 5.17 Color Stability of Liquid Detergent Composition at Mixing Temperature 60 °C

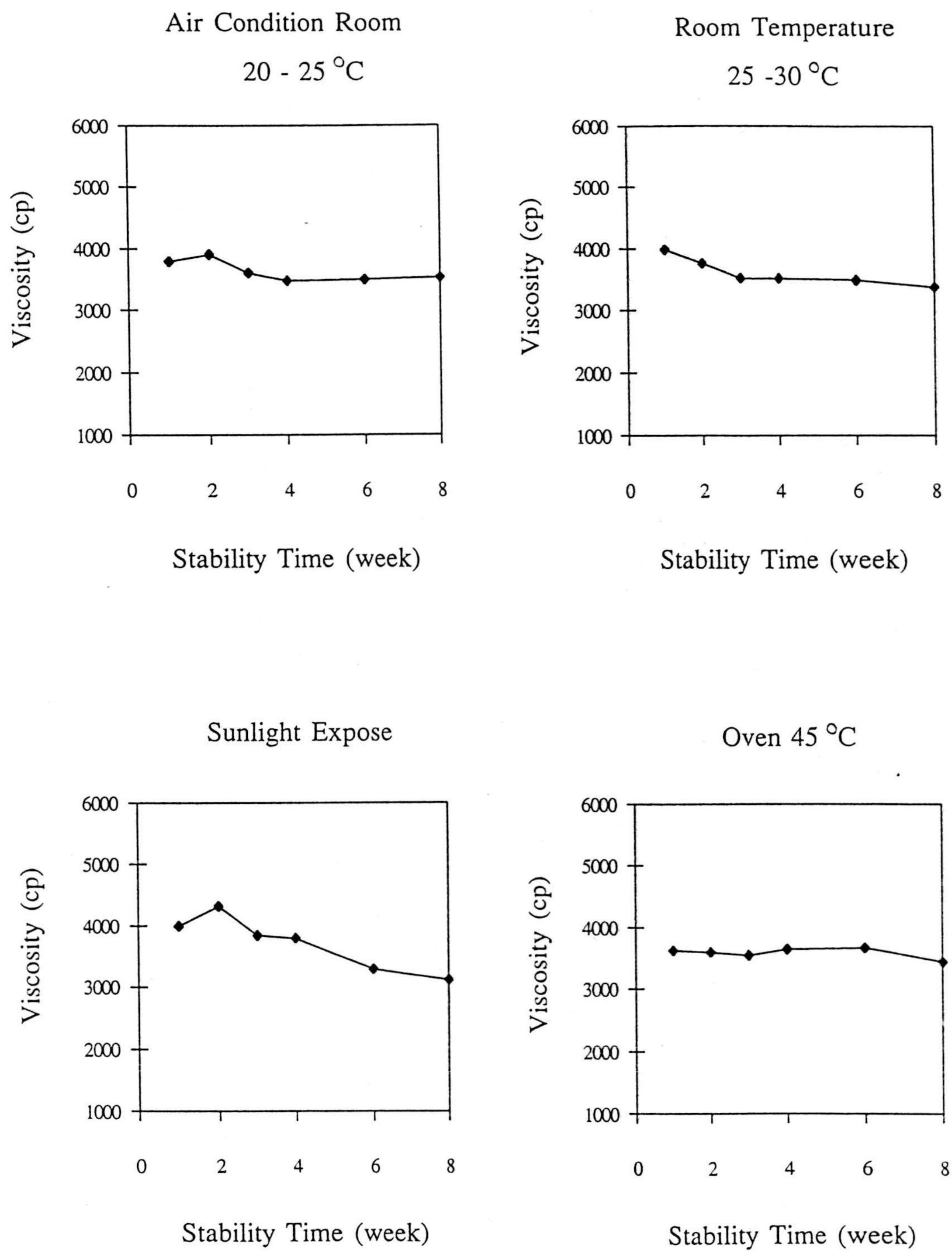


Figure 5.18 Viscosity Stability of Liquid Detergent Composition at Mixing Temperature 60 °C

Table 5.9 Characteristic of Liquid Detergent Composition from T.K. Agi Homomixer at 200 rpm

Typical Properties	Results
Active Matter (%)	15.2
Specific Gravity (30 °C)	1.009
Viscosity 30 °C (cp)	6400
pH (direct)	10.7

Table 5.10 Stability Test after 2 M of Liquid Detergent Composition From T.K.Agi Homomixer at 200 rpm.

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 3	5 -> 3
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	4175	4250	3725	4025
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

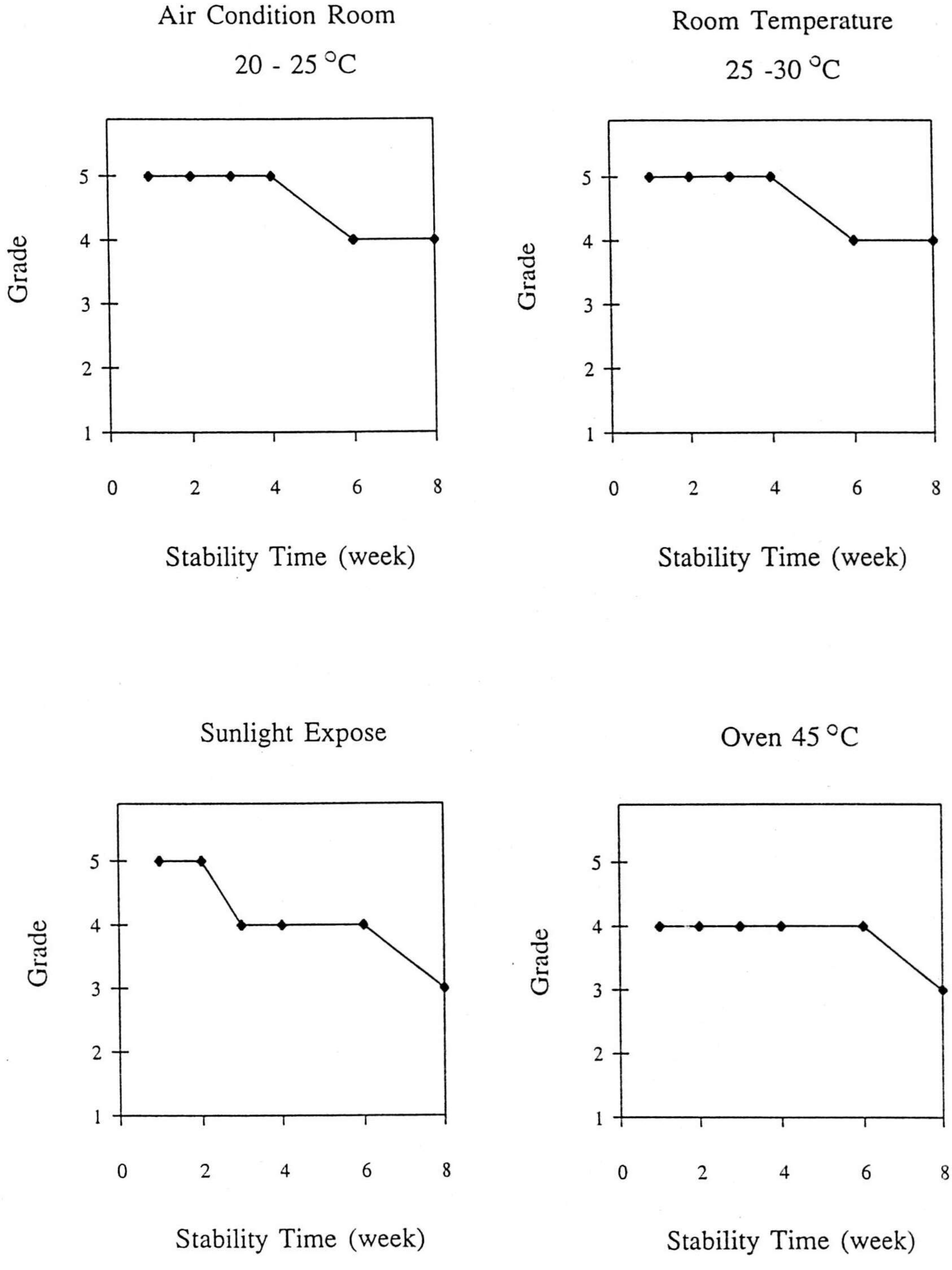


Figure 5.19 Odor Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 200 rpm

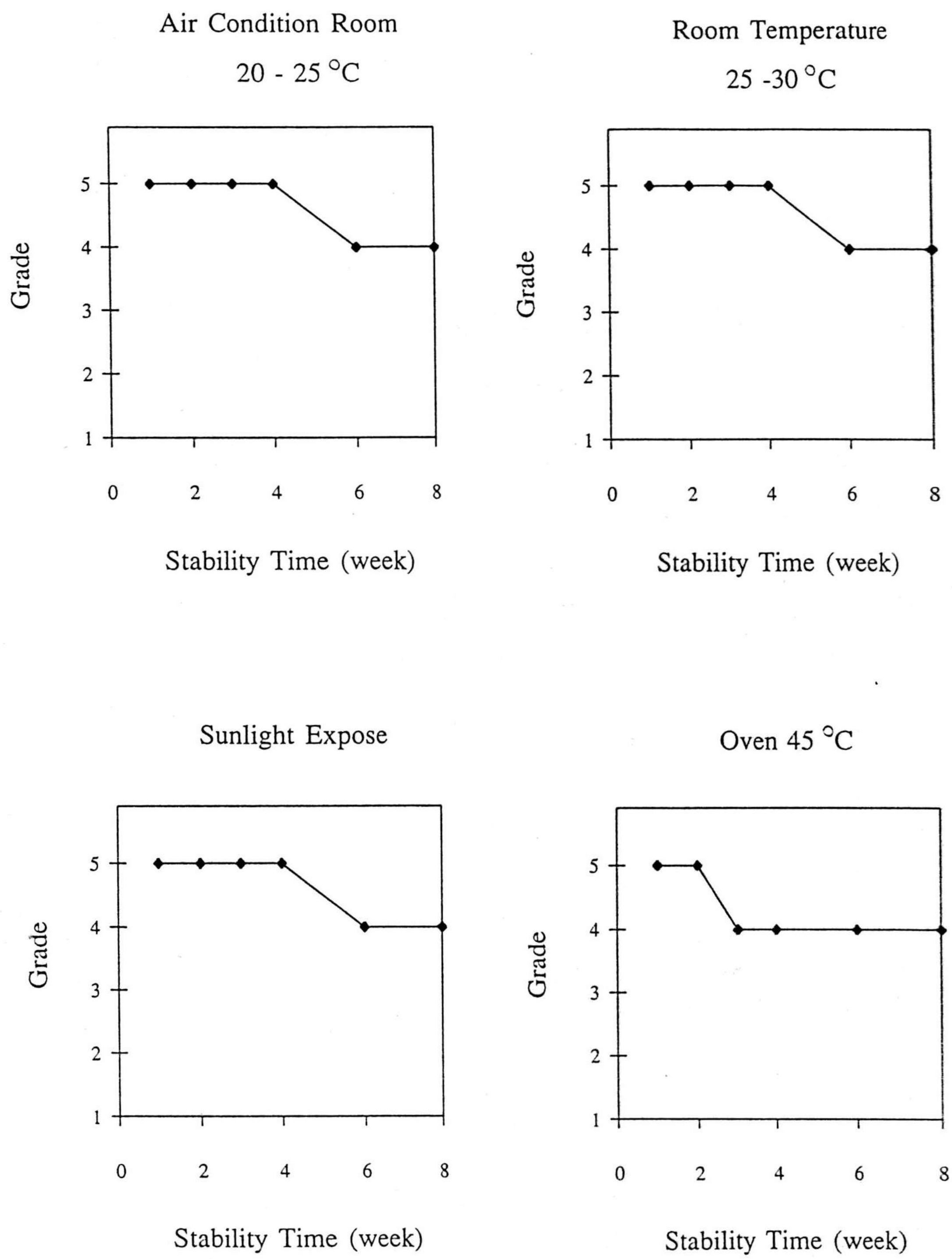


Figure 5.20 Color Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 200 rpm

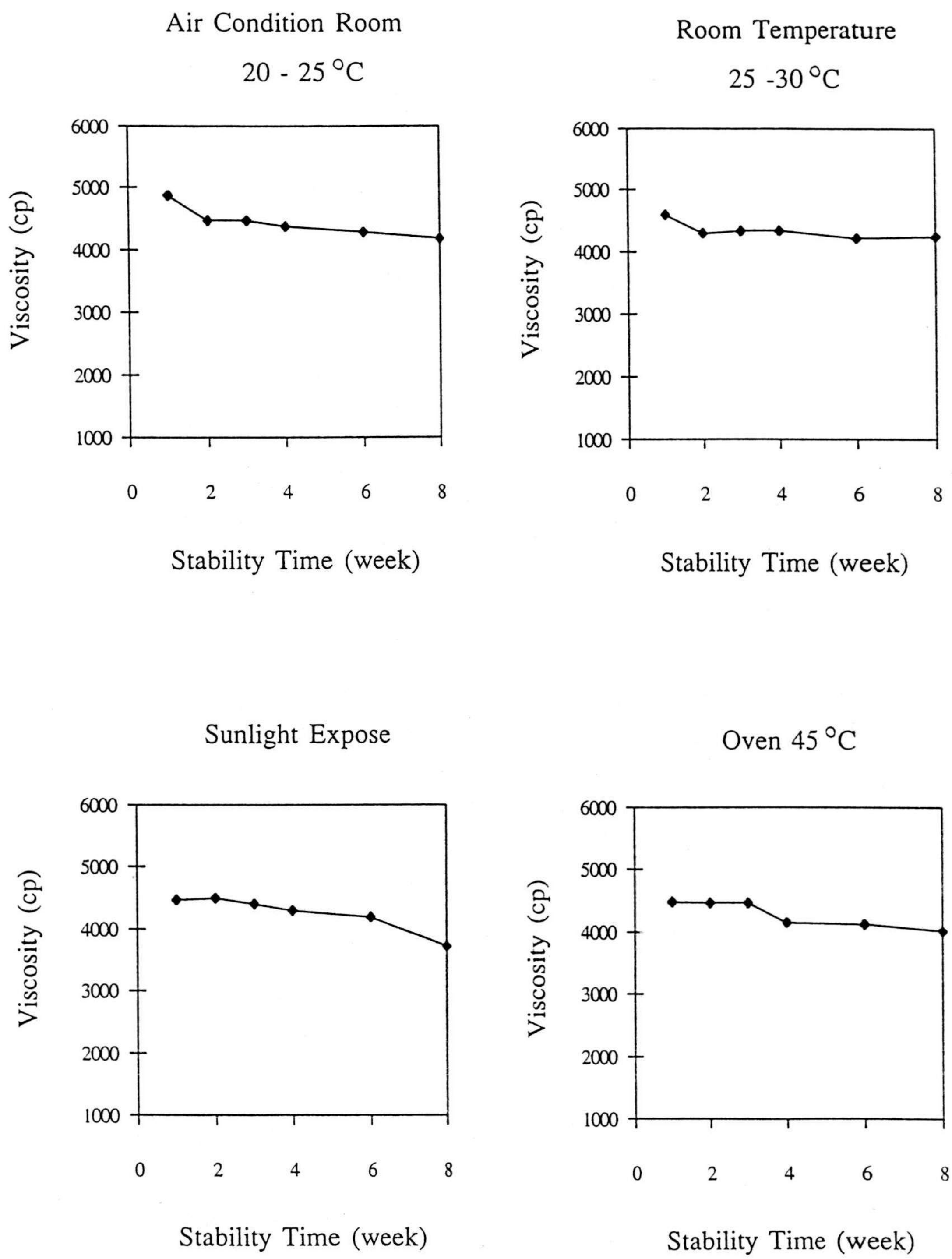


Figure 5.21 Viscosity Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 200 rpm

Table 5.11 Characteristic of Liquid Detergent Composition from T.K. Agi Homomixer at 320 rpm

Typical Properties	Results
Active Matter (%)	15.1
Specific Gravity (30 °C)	0.976
Viscosity 30 °C (cp)	6760
pH (direct)	10.6

Table 5.12 Stability Test after 2 M of Liquid Detergent Composition From T.K. Agi Homomixer at 320 rpm.

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 3	5 -> 3
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	4625	4620	4150	4300
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

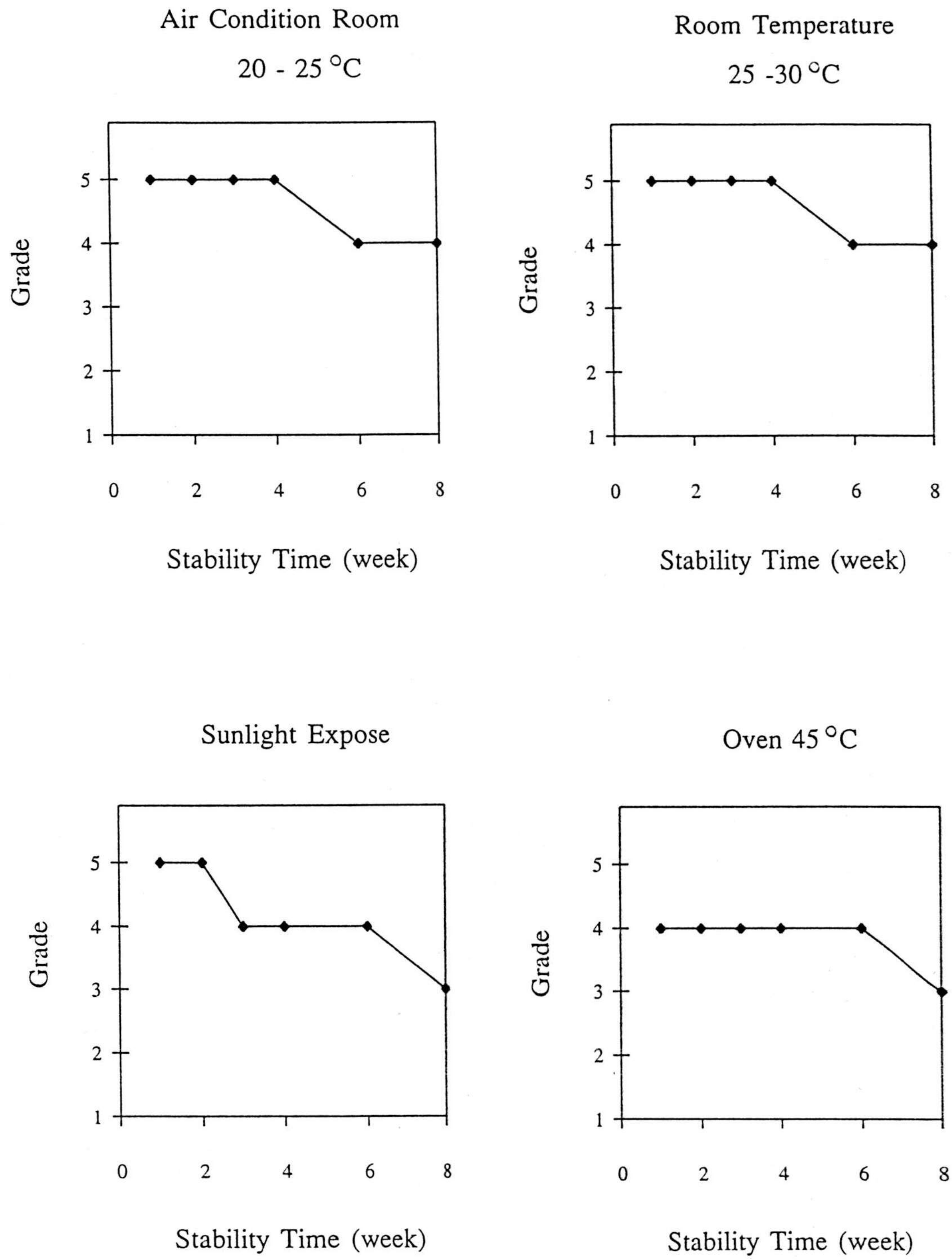


Figure 5.22 Odor Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 320 rpm

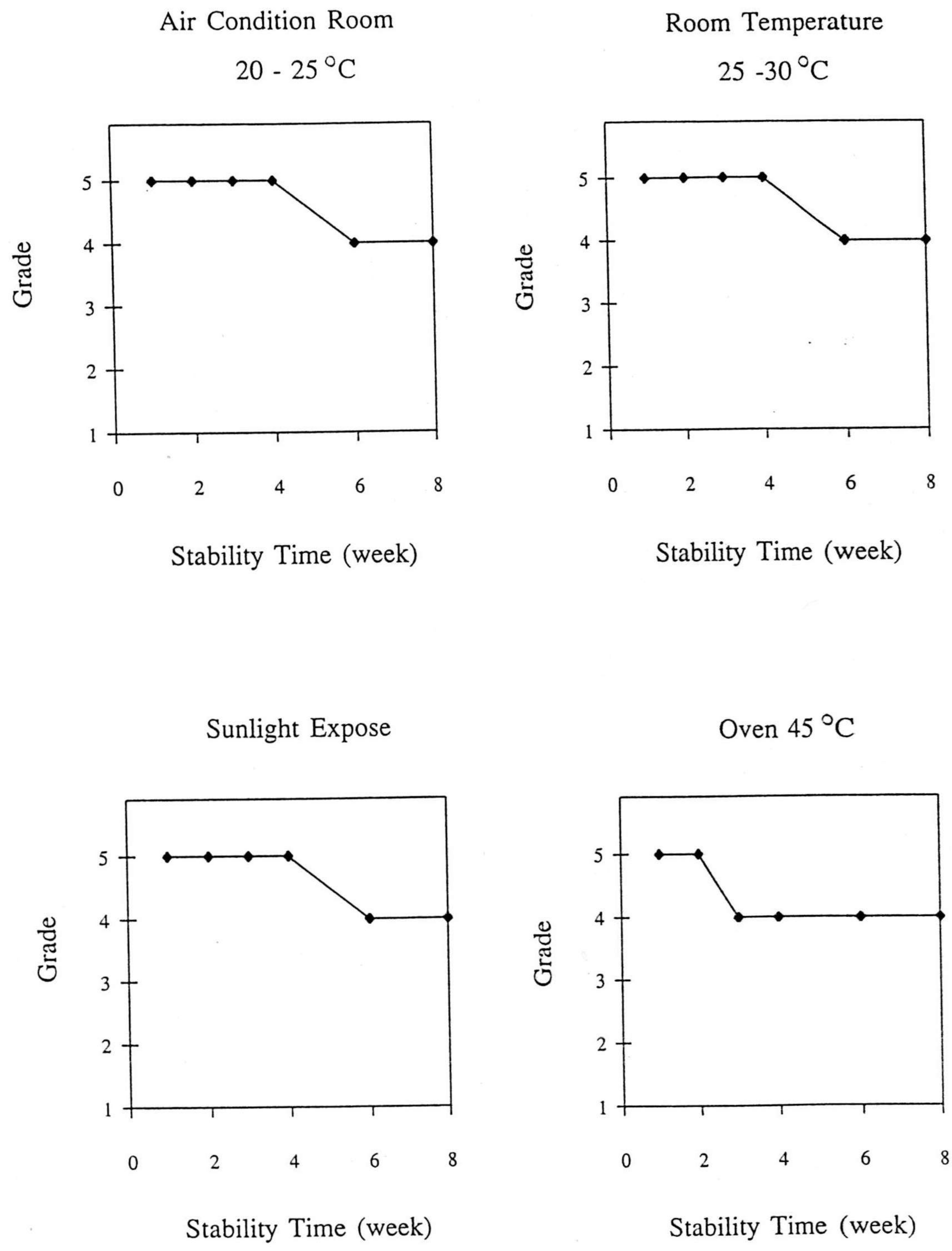


Figure 5.23 Color Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 320 rpm

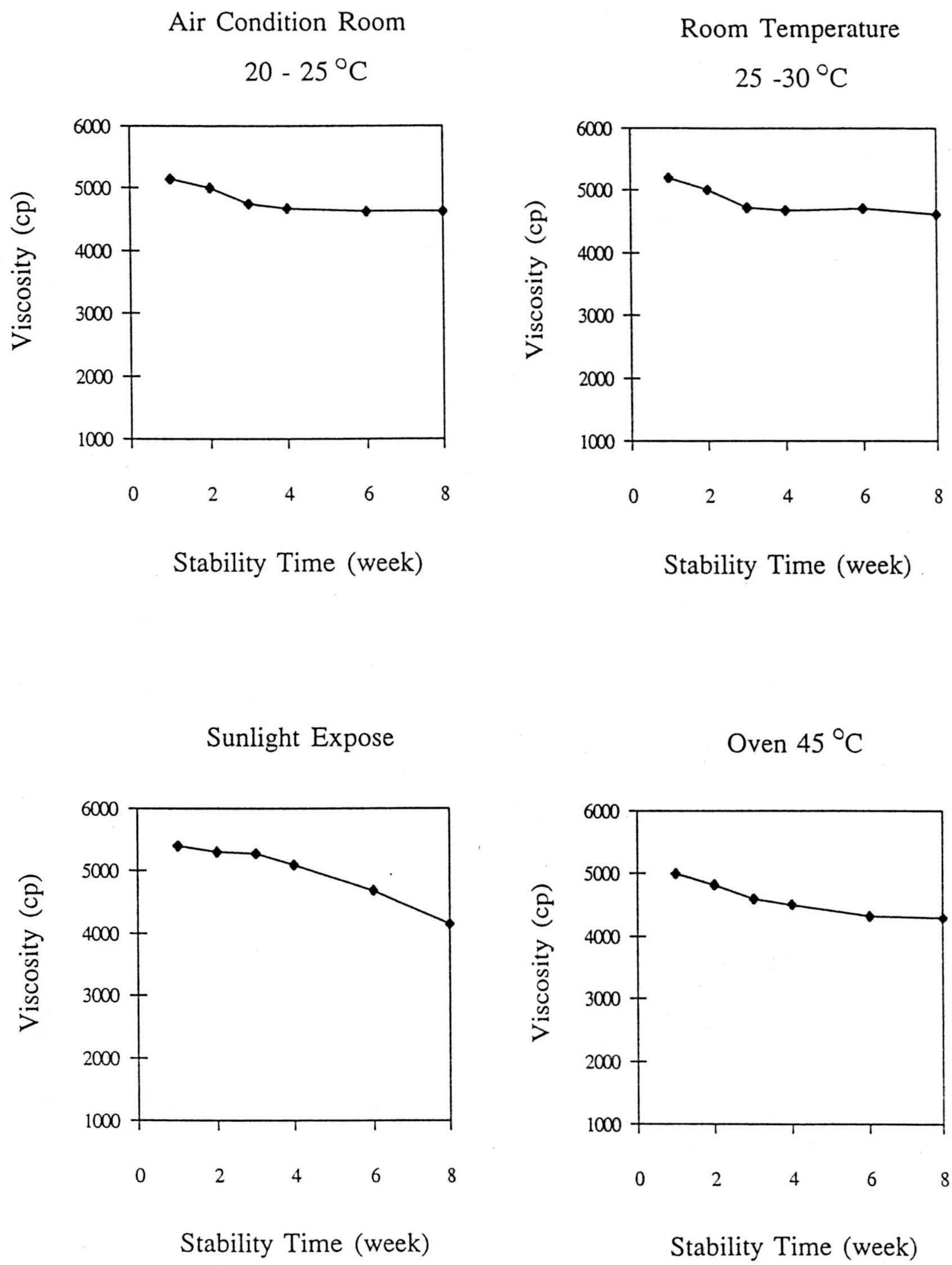


Figure 5.24 Viscosity Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 320 rpm

Table 5.13 Characteristic of Liquid Detergent Composition from T.K. Agi Homomixer at 430 rpm

Typical Properties	Results
Active Matter (%)	14.8
Specific Gravity (30 °C)	0.7465
Viscosity 30 °C (cp)	10800
pH (direct)	10.7

Table 5.14 Stability Test after 2 M of Liquid Detergent Composition From T.K. Agi Homomixer at 430 rpm.

Stability Item	Condition			
	Air Cond (20-25 °C)	Room Temp (25-30 °C)	Sunlight	Oven (45 °C)
Odor	5 -> 4	5 -> 4	5 -> 3	5 -> 3
Color	5 -> 4	5 -> 4	5 -> 4	5 -> 4
Viscosity (cp)	5750	5940	5590	5000
Separation	No Separation	No Separation	Slightly Separation	Slightly Separation
Freeze Thaw Test	No Separation	No Separation	No Separation	No Separation

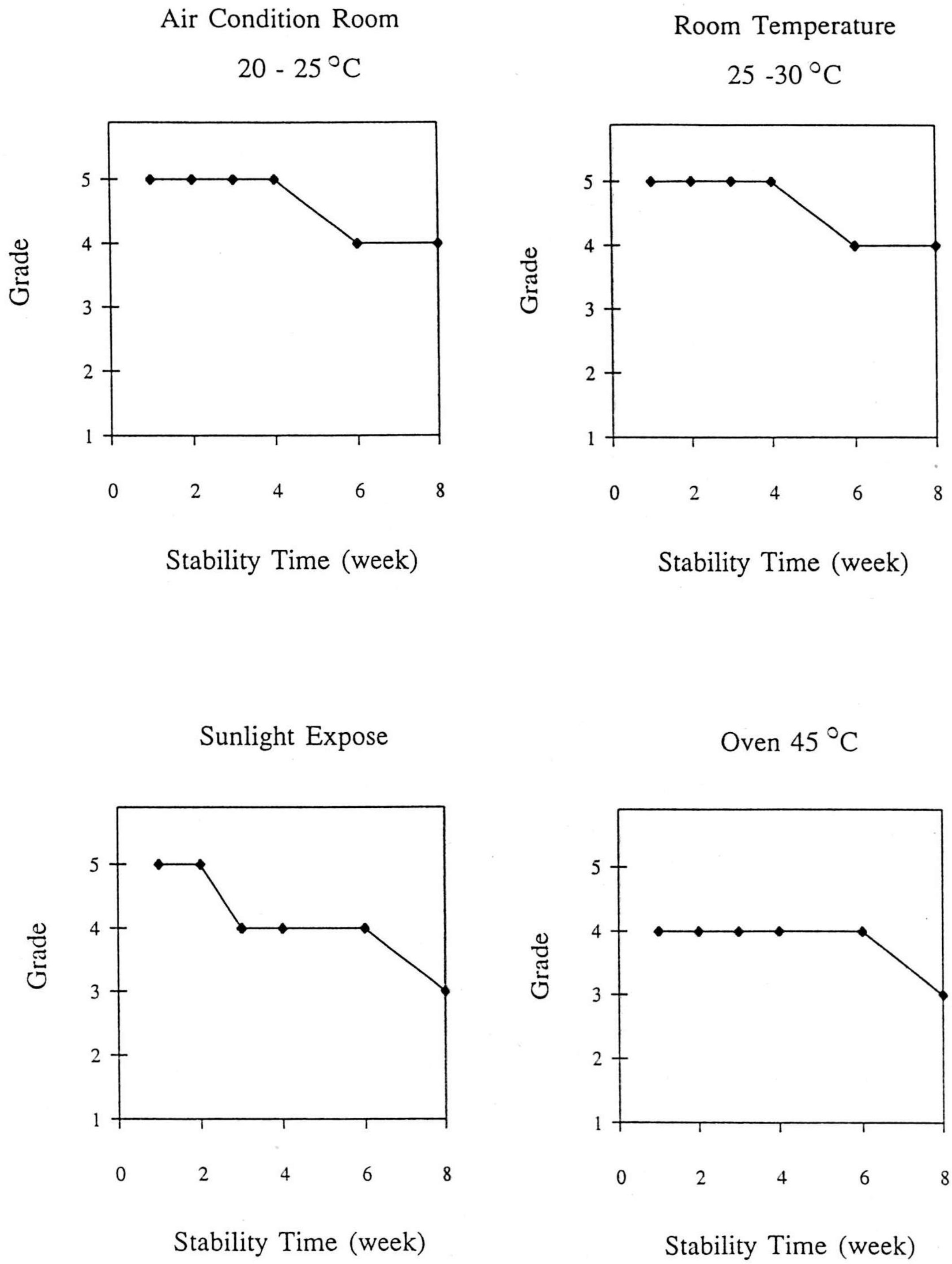


Figure 5.25 Odor Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 430 rpm

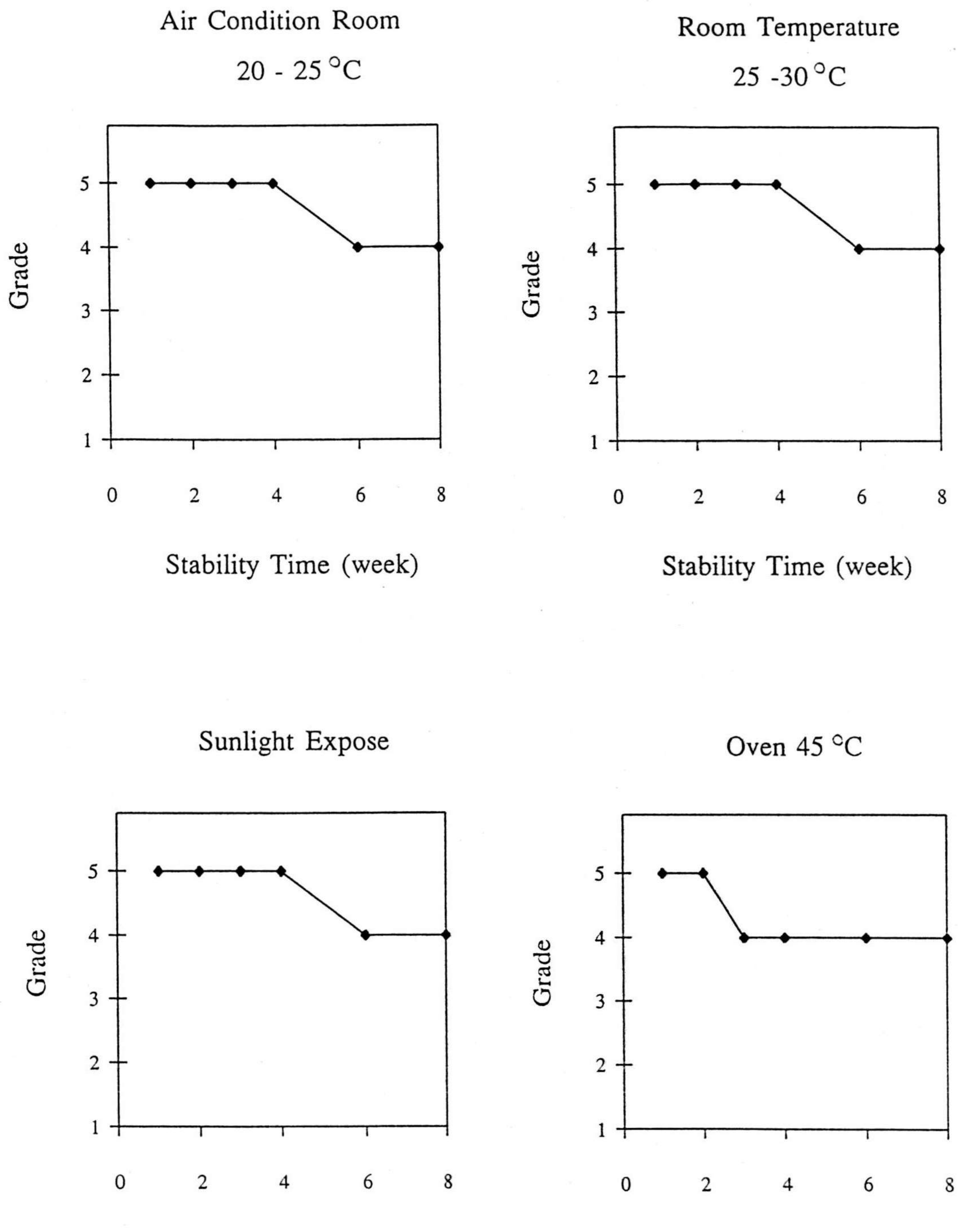


Figure 5.26 Color Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 430 rpm

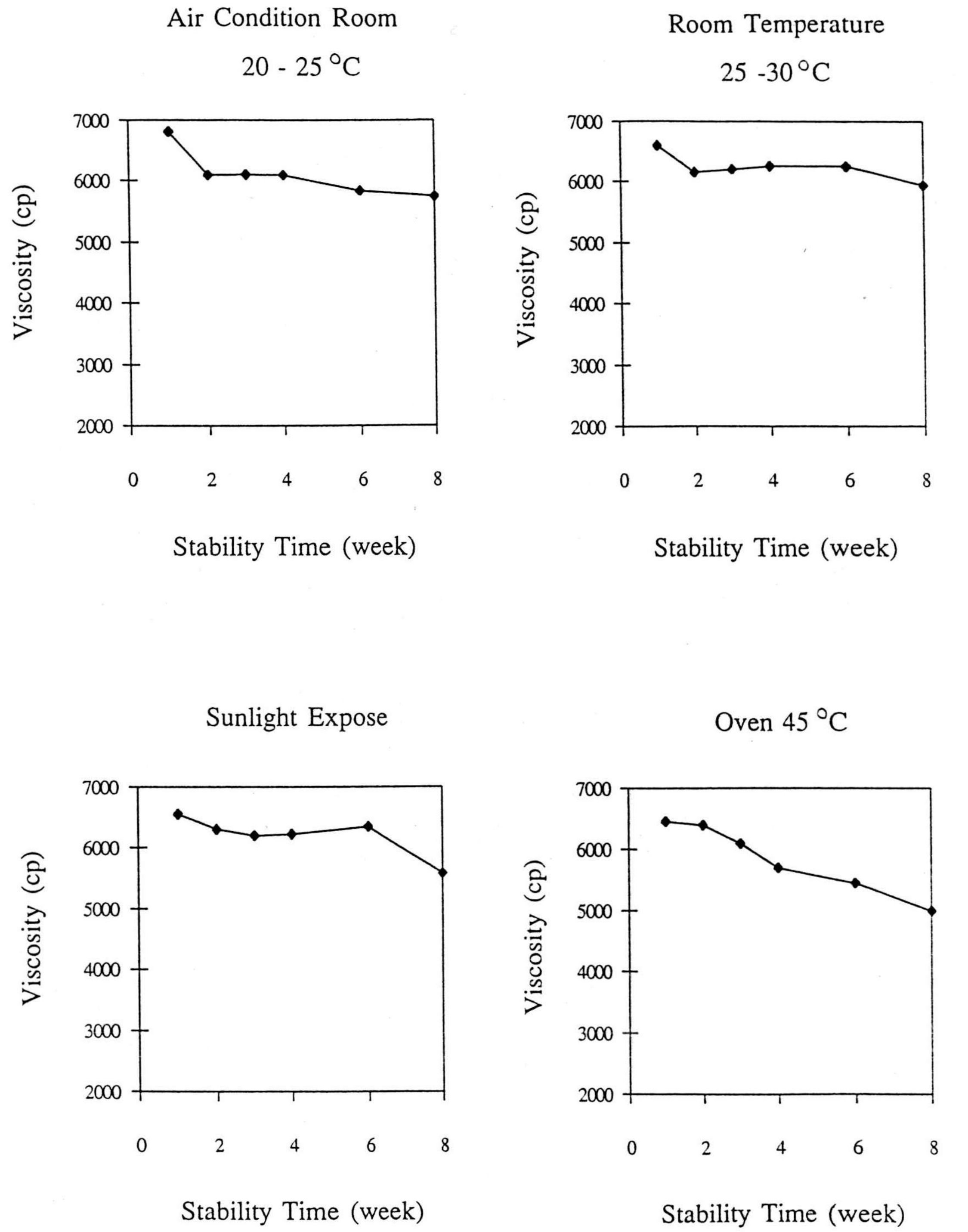


Figure 5.27 Viscosity Stability of Liquid Detergent Composition from T.K. Agi. Homomixer at Speed 430 rpm

Table 5.15 Typical Properties of Heavy Duty Liquid Detergent Built Formula in Thai Market

Typical Properties	Heavy Duty Liquid Detergent Built Formula			
	HDLD - SAMPLE	MARKET SAMPLE 1	MARKET SAMPLE 2	MARKET SAMPLE 3
Physical Properties				
Appearance	Opaque blue liquid	Opaque blue liquid	Opaque White liquid	Opaque White liquid
Viscosity (cp.)	2725	3450	1425	3450
Specific Gravity (30°C)	1.2058	1.1787	1.3266	1.1551
pH (direct)	10.8	10.2	10.7	9.8
Chemical Properties				
LAS (%)	12.2	12.1	23.4	12.8
AES (%)	2.8	2.2	3.8	1.7
STPP (%)	15.0	16.0	14.0	12.5
Sodium Carbonate(%)	5.0	5.8	10.1	2.1
Water Content (%)	62.6	63.9	38.7	69.2

Table 5.16 Performance Properties of Heavy Duty Liquid Detergent Built Formula in Thai Market

Performance Properties	Heavy Duty Liquid Detergent Built Formula			
	HDLD - SAMPLE	MARKET SAMPLE 1	MARKET SAMPLE 2	MARKET SAMPLE 3
Detergency(%)	111	92	96	102
Foaming (%)	114	103	115	103
Whitening (%)	101	102	104	100