Purification of an acrylic IOL dye additive by a solvent washing process – Effect of precipitation temperature

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Introduction: "UV Chromophore" (**Figure 1**) is an ultraviolet-light blocking additive used in soft acrylic materials for ophthalmological application. In this study, we evaluated the purity of UV Chromophore purified by a solvent washing process. After dissolving the UV chromophore in hot methanol, precipitate fractions were collected at different cooling temperatures, and the properties of these precipitates were evaluated.

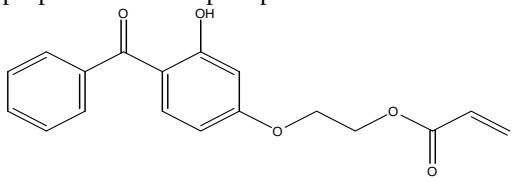

Figure 1. Chemical structure of UV blocking additive CAS#: 16432-81-8; Empirical formula: $C_{18}H_{16}O_5$;

This study summarizes the purity-related results for the UV chromophore after purification by a solvent (methanol) washing process, especially assessing the effect of precipitation temperature on the properties by melting point and HPLC studies. Melting range and HPLC area percent were used for comparing the fractions regarding purity.

Material and Methods: UV Chromophore purchased from vendor was used directly in this study. 100 grams of raw UV chromphore was dissolved in hot methanol (60°C) under magnetic stirring. After half hour of stirring, a brown color chunk solid ball was clearly visible in the container. This ball was collected after filtration and was considered as impurity (as indicated by DSC data later) of the UV chromophore. The solution after filtration was collected, stored in the beaker and placed in the hood with glass cover for 24 hours. After 24 hours, a lot of precipitates were observed in the container. These precipitates were collected again with filtration and put in the 60°C oven for 72 hours to remove any solvent residues. Precipitate collected at this stage was labeled as room temperature precipitate. Following the same procedures, precipitates were also collected at 6°C (refrigerator) and -3°C (freezer) for evaluation.

These precipitates were evaluated by melting points (both capillary and DSC) and HPLC.

Results: The yield of UV Chromophore samples collected at room temperature, 6°C and -3°C were summarized in **Table 1** as below:

Initial wt. / yield	Room Temperature	6°C	-3°C	Undissolved Impurity
100 g	89.6 g	4.3 g	1.0 g	1.4 g
%	93.0%	4.5%	1.0%	1.5%

Table 1. Yield of precipitated dye at different precipitation temperatures

Results of melting temperatures of precipitates collected at different temperatures measured by both capillary method and DSC are summarized in **Table 2**

UV Chromophore	Capillary Melting Point, °C				DSC Melting Point, °C
	Automatic Determination		Manual Determination		
	Onset	Clear	Onset	Clear	
Raw Material	81.8 ± 0.1	83.0 ± 0.5	82.0 ± 0.1	83.1 ± 0.1	81.0 ± 0.3
Room Temp ppt	82.5 ± 0.1	83.4 ± 0.1	82.6 ± 0.1	83.5 ± 0.1	82.5 ± 0.3
6°C ppt	82.4 ± 0.1	83.1 ± 0.1	82.6 ± 0.1	83.1 ± 0.1	81.5 ± 0.3
- 3°C ppt	81.8 ± 0.2	82.7 ± 0.2	82.0 ± 0.1	82.9 ± 0.0	80.4 ± 0.5

Table 2. Comparison of Capillary and DSC Melting Point Results

Melting temperature was higher for samples collected at high temperature. Melting points of undissolved brown, rigid balls collected at the beginning of the washing process were measured by DSC and in the range of 75.2 ~ 76.7°C indicating a significant level of impurity. Due to its very different appearance, no further test was done on this impure material.

Purity of UV Chromophore was determined by HPLC area percent for these precipitates, as summarized in **Table 3**.

Sample	% Percent Purity			
	Test 1	Test 2	Test 3	Average
Raw material	95.6	95.7	95.7	95.7
Precipitates at RT	98.0	98.0	98.0	98.0
Precipitates at 6°C	96.1	96.1	96.1	96.1

Table 3. Percent Purity Results by HPLC

As shown from **Table 3**, the purity values by HPLC area percent were higher for precipitates collected at higher temperatures.

Conclusions: The purity of solvent-wash precipitates of UV Chromophore depended on temperature of precipitate collection. Precipitates collected at higher temperatures (room temperature here) showed higher purity as indicated by the higher melting point and HPLC percent values as compared to precipitates collected at lower temperatures (6°C and -3°C). Methanol washing was generally effective at improving the raw material purity, but precipitate collection temperature was found to also be important.