Physical Vapor Growth of Rubrene

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- **Abstract**

Rubrene is a novel organic semiconductor with many interesting electronic properties. Among organic semiconductors, rubrene has the highest carrier mobility, with values reaching 40 $cm^2/(V\ S)$.[5] These electronic properties, however, are only applicable for crystallized and pure forms of rubrene.[4]

Physical vapor transport of rubrene is capable of purifying and crystallizing rubrene powder. This is accomplished by heating the impure sample and subjecting the sublimated vapors with a constant flow of Ar gas along a temperature gradient. We report efforts to build and test a physical vapor growth system for rubrene.

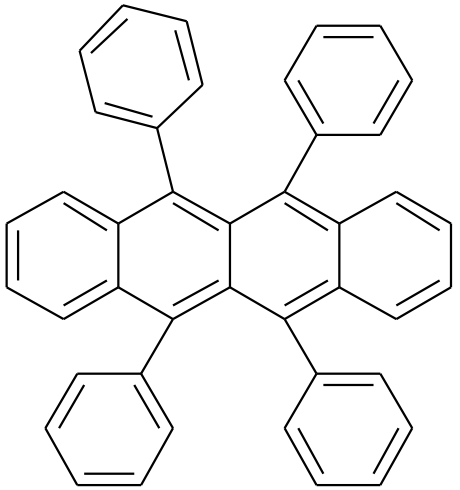


Figure 1: Rubrene ($C_{42}H_{28}$)

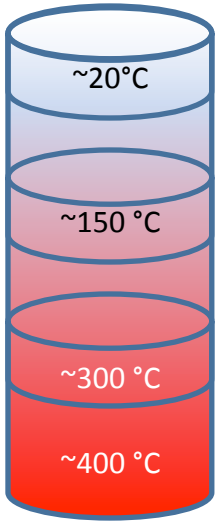
- **Introduction**

Physical vapor transport (PVT) is a technique used to separate materials of different volatility. The process is identical to fractional distillation, except that distillation depends on the transitions between liquid to gas, while PVT relies on the transitions between solid to gas.

For either technique, different zones along the column are maintained at different temperatures, seen in Figure 2.

Rubrene, the compound of interest, sublimes at ~280 °C[1]. Its impurities are assumed to not have the same temperature of sublimation. Therefore, a purer form of rubrene grows at the particular temperature zone

A)



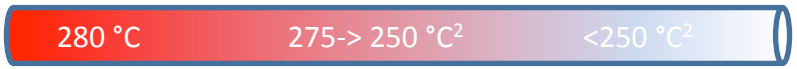
Gas Here

Gasoline Here

Diesel Here

Crude Oil is heated

B)



Crude Rubrene is heated at sublimation temperature

Rubrene Here

Impurities here

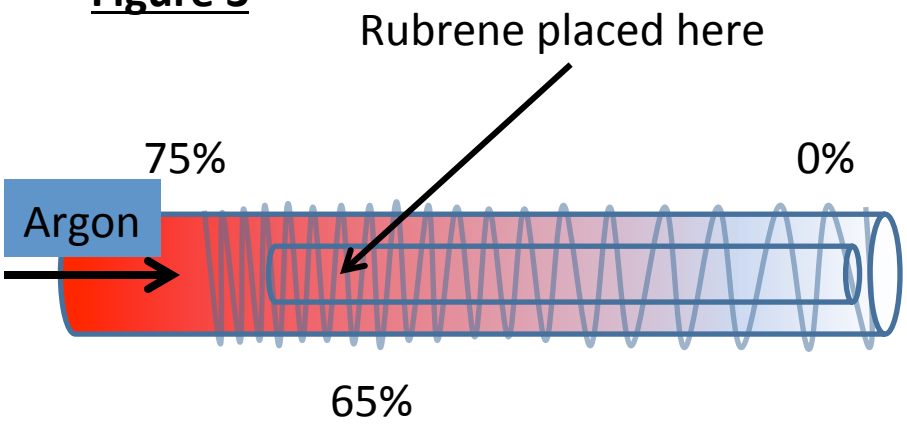
- **Experimental Methods**

Nichrome wire was coiled around a large borosilicate tube densely, with no contact between coils, starting at 75% of the tube's length. As the coils advance to the opposite end of the tube, the density of coils was decreased (Figure 3).

The temperature as a function of distance from the loose coiled end of the tube (temperature profile) was then adjusted either by recoiling the tube, adjusting the Variac or by wrapping fiber glass insulation in specific areas. These adjustments should lead to roughly 280°C as a peak temperature at 65% of the tube's length and should linearly decrease to 200°C at 25% of the tube's length.

Three different temperature probes were evaluated in order to measure the temperature consistently and over long periods of time: thermistor, platinum resistor, and thermocouple wire.

Figure 3



- **Experimental Methods**
  - **Miscellaneous Calibration**

    A method was initially desired to find ways to record the temperature overnight. Initially, thermistors were calibrated with a hot sand bath parallel with a thermocouple. The same was performed with platinum resisters. Overnight measurements were recorded with LabView.

  - **Placement of Thermistors**

    Holes were created into glass tubes for the thermistors/resistors by heating a small portion of the tube with a torch, closing one end of the tube and blowing air into the other side. The heat creates a temporary structural weakness and a glass bubble appears, then pops, which then creates the desired hole. The smaller the area that is heated, and the cooler it is, the smaller the hole.

Figure 4: Temperature Profile

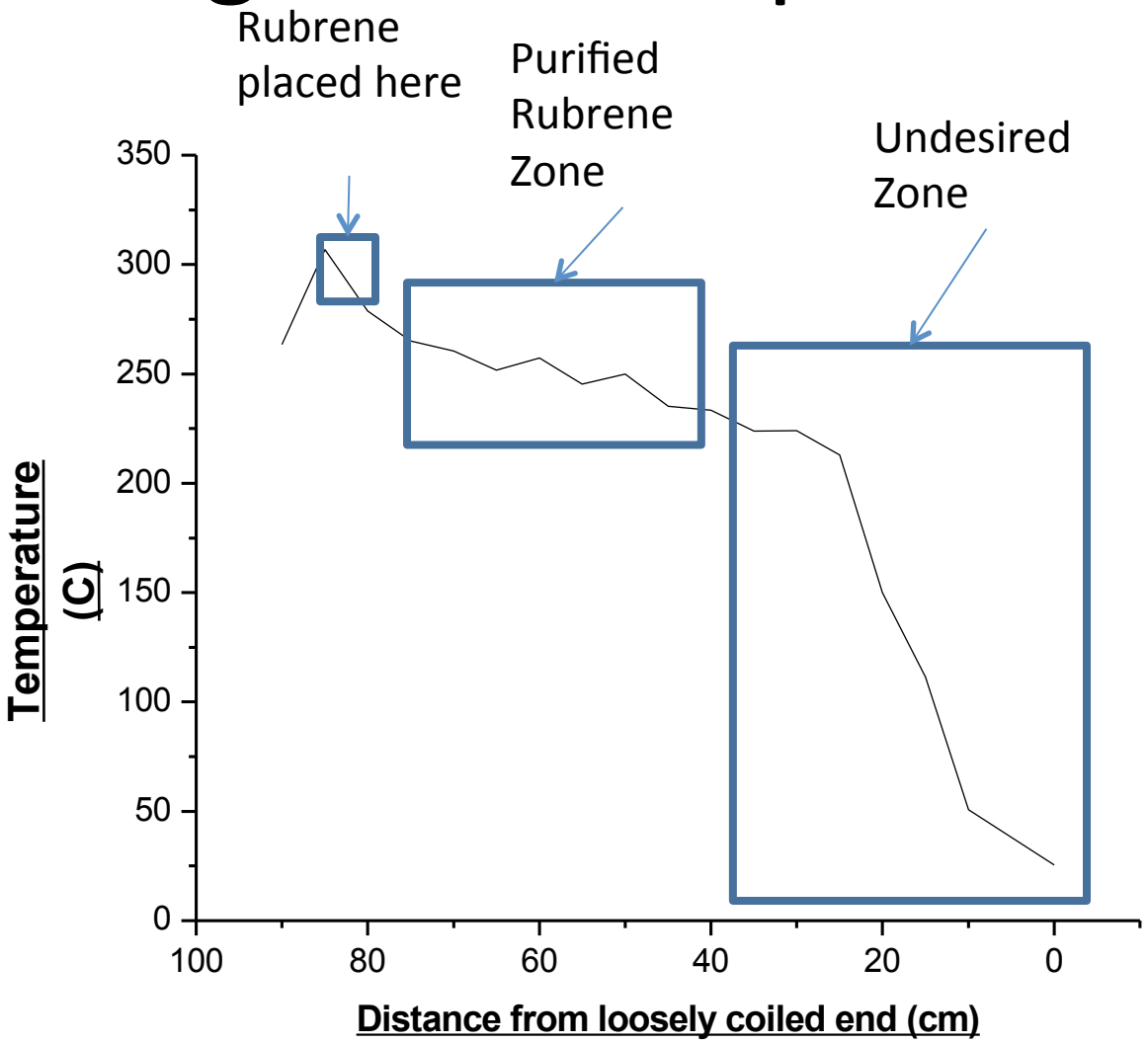


Figure 4: Graph of temperature along column. While the areas between 275°C to 225°C has some kinks, the shape is largely linear, as desired.

Figure 5: Single Spot Overnight Temperature Measurement

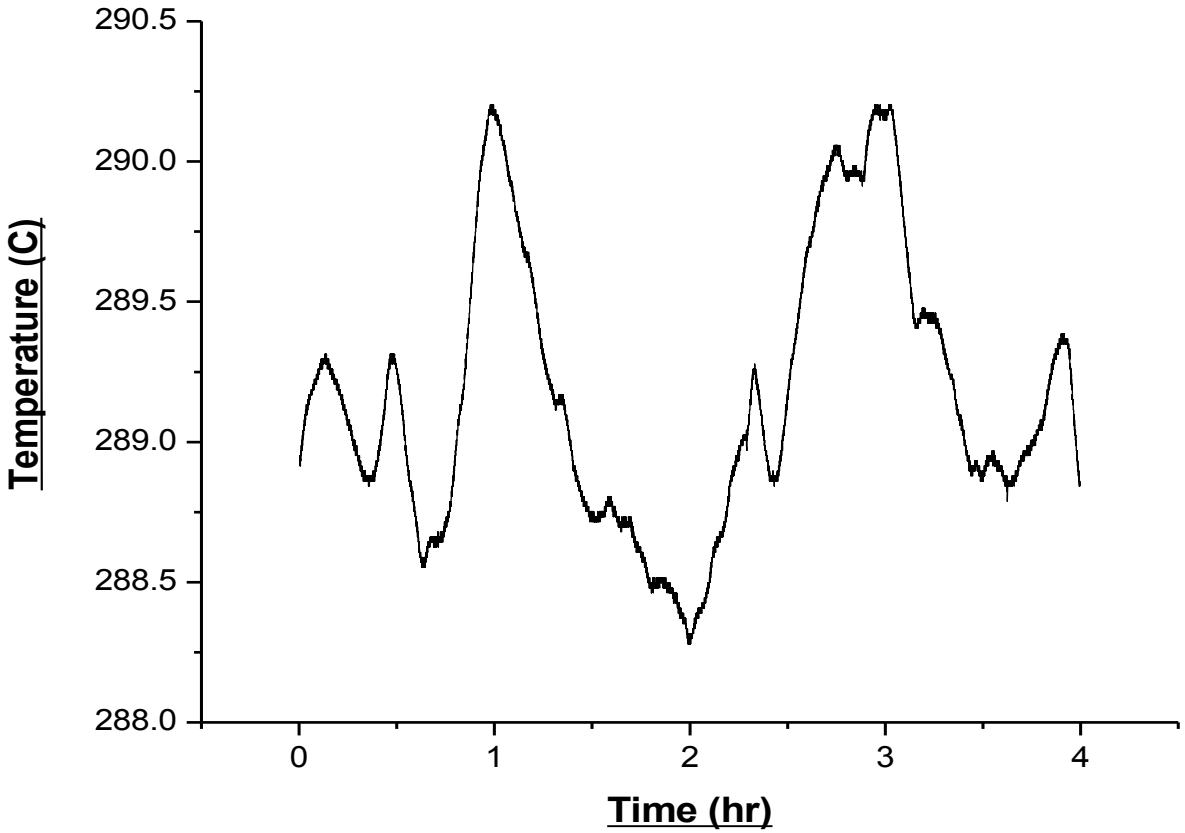


Figure 5: Graph of temperature vs. time at one location within the furnace. As one can see, there is some variance.

- **Discussion**
  - **Purification**

Teflon decomposes at temperatures above 200°C.[3] This can be an issue if we desire to purify rubrene, as methods of purification should not add more contaminants. Perhaps a different substrate is needed.

If possible it is recommended to use as large of a gauge as possible so that coils don't have to be so dense (large gauge -> smaller diameter -> more resistance -> more heat -> lower settings on Variac -> more control over temperatures).

Rubrene appears to have a liquid phase as well, at temperatures above 315°C a dark red gelatin can be formed after overnight runs and spread over the Teflon sheet.

- **Discussion**
  - **Thermistors/Resistors**

The thermistors originally used were robust and did not break easily. However, they did not accurately measure the temperature above 250°C very well.

The platinum resistors measured the temperature very well but were very fragile and easily prone to break.

In the end, a thermocouple seemed to be the most robust and accurate compromise between the two and was used in the overnight measurements.

- **Oxygen**

  Oxygen oxidizes rubrene and this oxidized form is a contaminant. The argon gas acts as both a carrier gas (to move the rubrene in one direction) and also to remove the oxygen from the tube. However it cannot remove all the oxygen from the tube.

  To alleviate this one must leave the argon running for some amount of time before inserting rubrene. It is not known how long one must do this.

- **Temperature Variance**

  The variance in figure 5 is not especially disturbing. We expect some temperature fluctuations as measurements occur in a basement overnight.

Conclusion

While the apparatus is more or less functional there are still some minor interferences to be fixed. While teflon and oxygen are issues that can probably be resolved there are still issues regarding the temperature variance that need to be addressed before serious measurements of the properties of these crystals can be performed.

Acknowledgements

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Citations

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