Tech Bulletin

Thermal Analysis of DT-420

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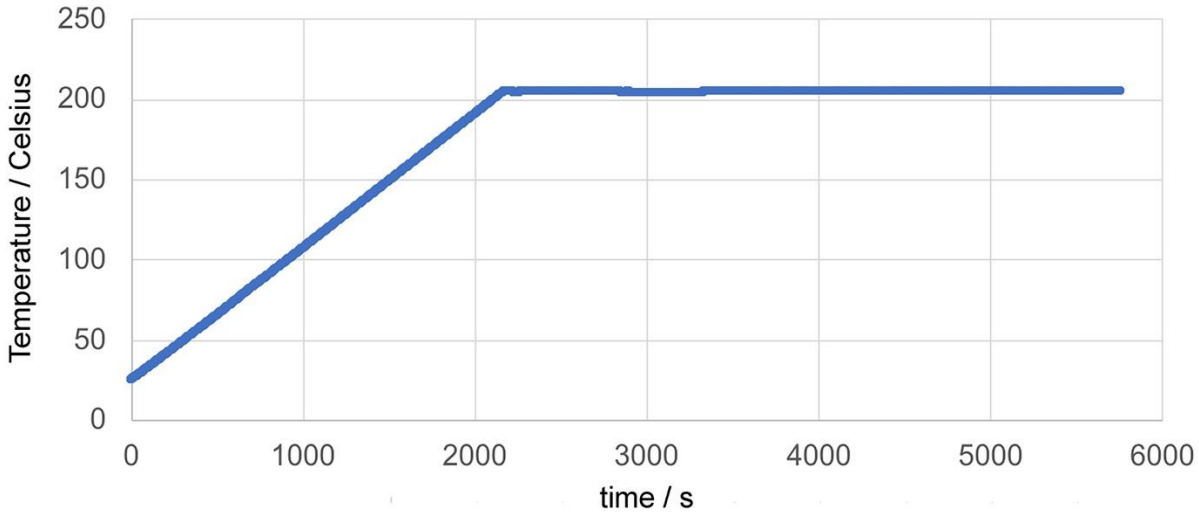
To model the behavior of DT-420 on steel during thermal conditions typical for mold release agents, we ran three consecutive heating cycles with a coating of DT-420 on steel. The first run was performed immediately after DT-420 was applied, while the second and the third runs were performed using the coating applied in run 1. The DT-400 series of coating compositions (including the DT-420 product featured here) are covered under U.S. 9,856,400 issued January 2, 2018. Coatings formed with the DT-400 series are the subject of pending U.S. Patent Application No. 15/820,853 filed November 22, 2017.

Figure 1 shows the thermal conditions identical during all three consecutive runs. The temperature is linearly increased to 205 ºC (400 ºF) during 2150 s and then remains constant for 1 h (3600 s).



In Figure 2, the loss of mass of the DT-420 coating on steel as a function of heating is shown, as determined by Thermogravimetric Analysis (TGA). DT-420 lost mass continuously during run 1. Therefore, the chemical composition of the gases emitted from the DT-420 coating were analyzed by mass spectroscopy.

As shown in Figure 3, at 500 s (65 ˚C/150 °F), a mixture of isopropyl acetate and aliphatic hydrocarbons is evaporated while the coating is formed. This finding is consistent with the formulation of DT-420.

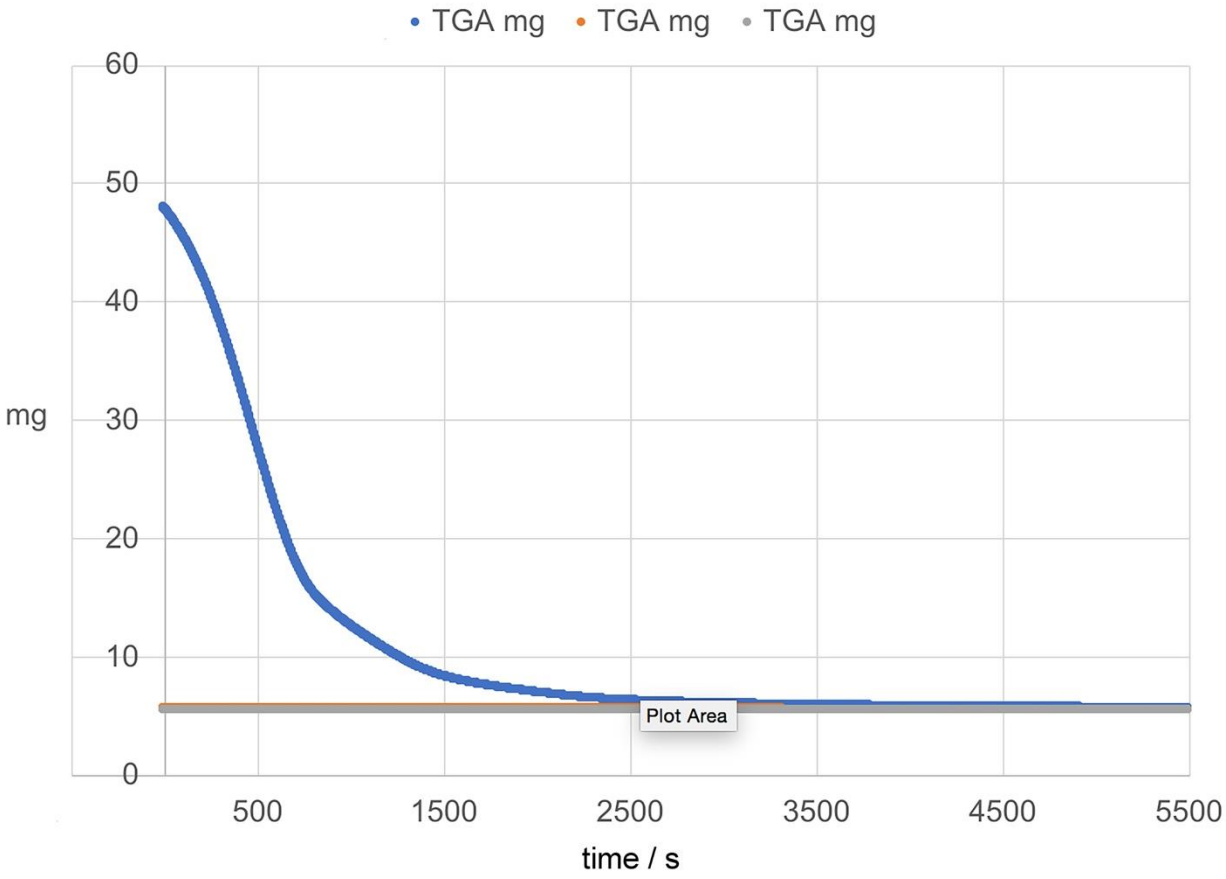


Figure 2: Thermogravimetric Analysis (TGA) of DT-420 on steel. Three consecutive runs (blue: first run; orange: second run; gray: third run) with the same coating were performed according to the temperature profile shown in Figure 1.

As shown in Figure 4, at 1000 s (108 °C/226 ºF), a mixture of isopropyl acetate and aliphatic hydrocarbons is evaporated. Compared to the chemical composition of the vapors emitted from DT-420 at 500 s, at 1000 s a relative enrichment of isopropyl acetate is observed. Again, this agrees with the formulation of DT-420.

However, at 1500 s (150 °C /302 °F), the composition of the emitted gases significantly changes. At that temperature, a mixture of isopropyl acetate and ammonia (NH_3) is detected. The emission of ammonia during the thermal curing of DT-420 is typical for the chemistry of polysilazanes. See U.S. 9,856,400 at col. 10, ll. 6–24.

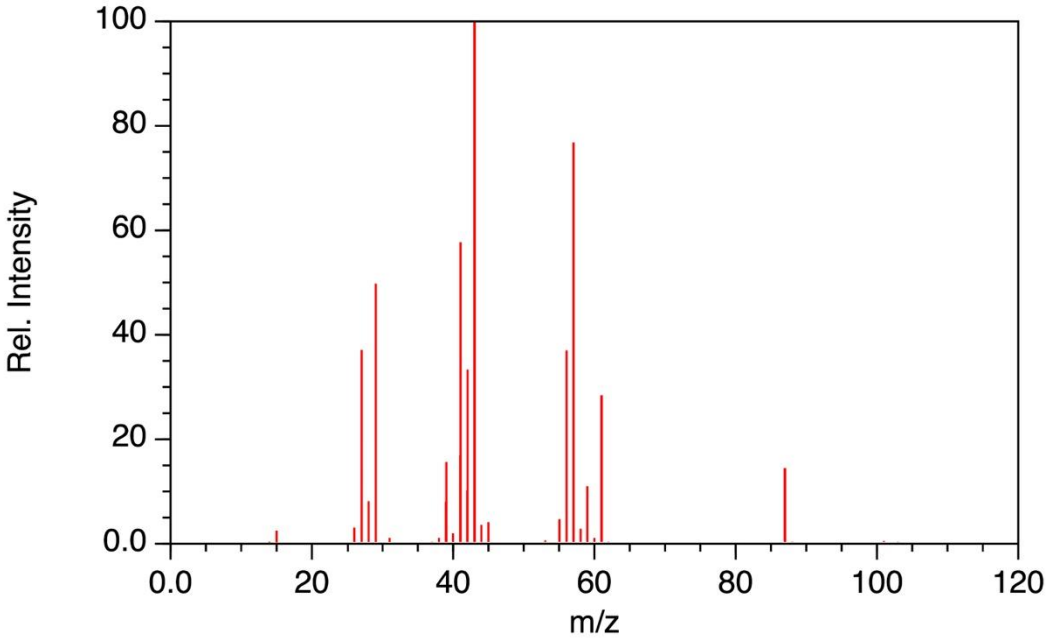


Figure 3: Mass spectrum of the gas phase above DT-420 (run 1, 500 s). The mass spectrum is consistent with a mixture of isopropyl acetate and aliphatic hydrocarbons (reference: https://webbook.nist.gov/chemistry).

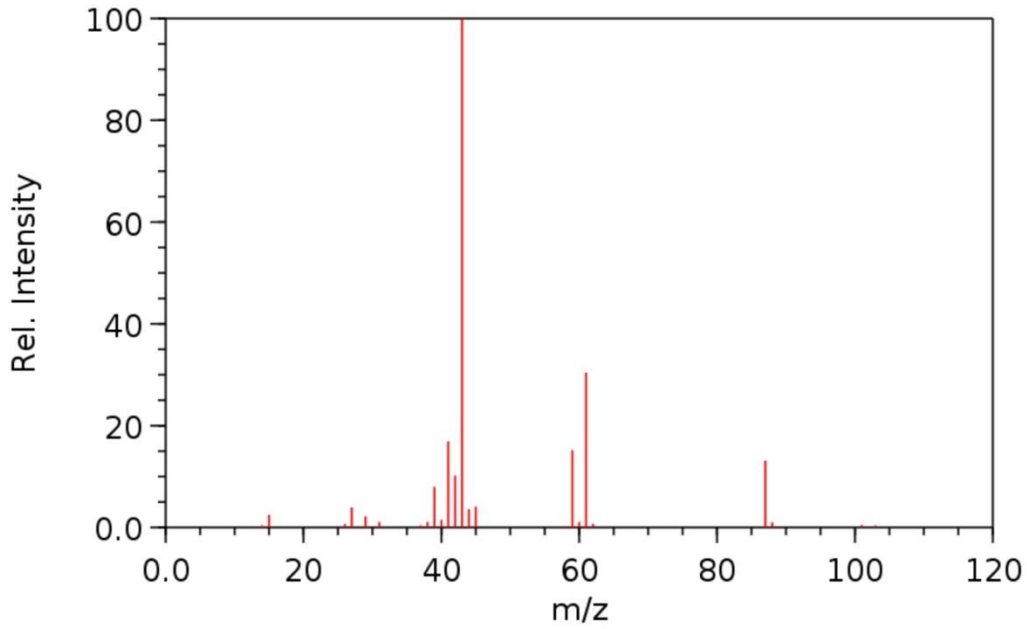


Figure 4: Mass spectrum of the gas phase above DT-420 (run 1, 1000 s). The mass spectrum is consistent with a mixture of isopropyl acetate and aliphatic hydrocarbons (reference: https://webbook.nist.gov/chemistry).

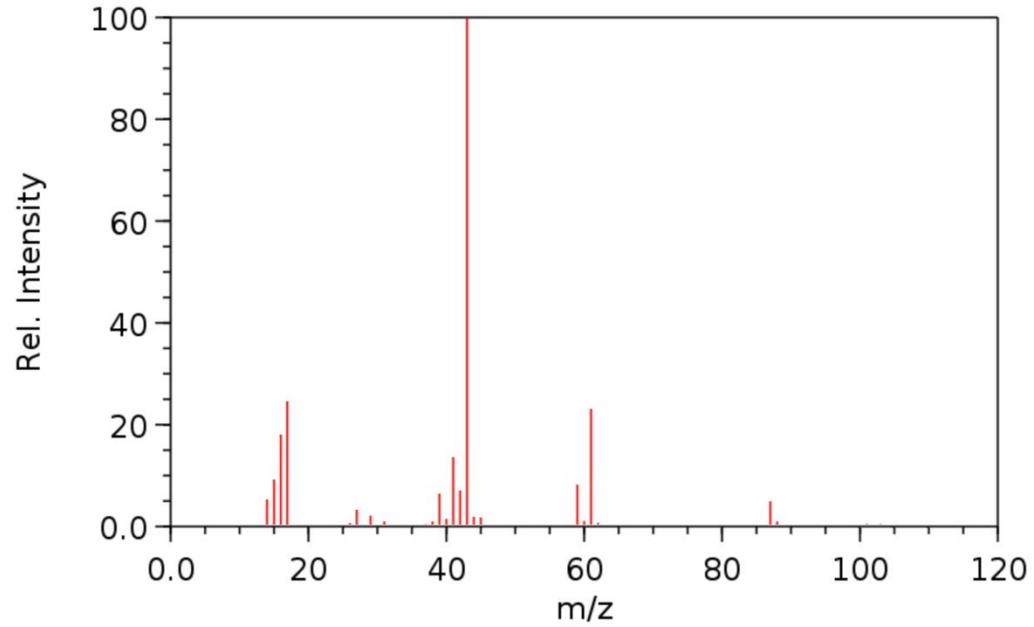


Figure 5: Mass spectrum of the gas phase above DT-420 (run 1, 1500 s). The mass spectrum is consistent with a mixture of isopropyl acetate and ammonia (cluster at masses 17, 16, 15 and 14). (reference: https://webbook.nist.gov/chemistry).

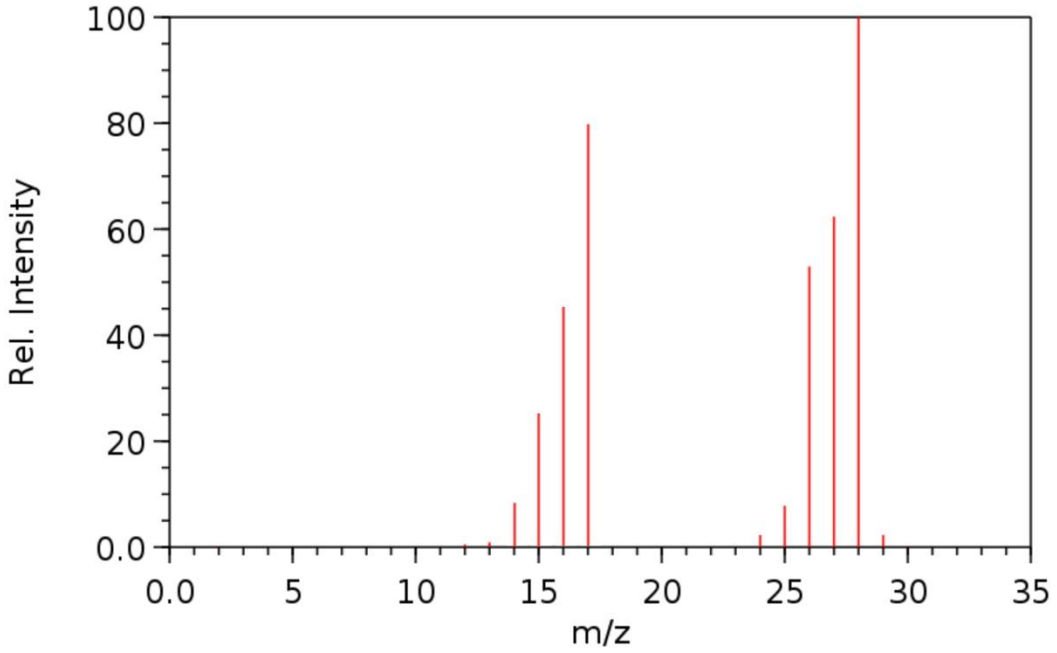


Figure 6: Mass spectrum of the gas phase above DT-420 (run 2, 3500 s). The mass spectrum is consistent with a mixture of ethylene (and potentially other hydrocarbons) and ammonia (reference: https://webbook.nist.gov/chemistry).

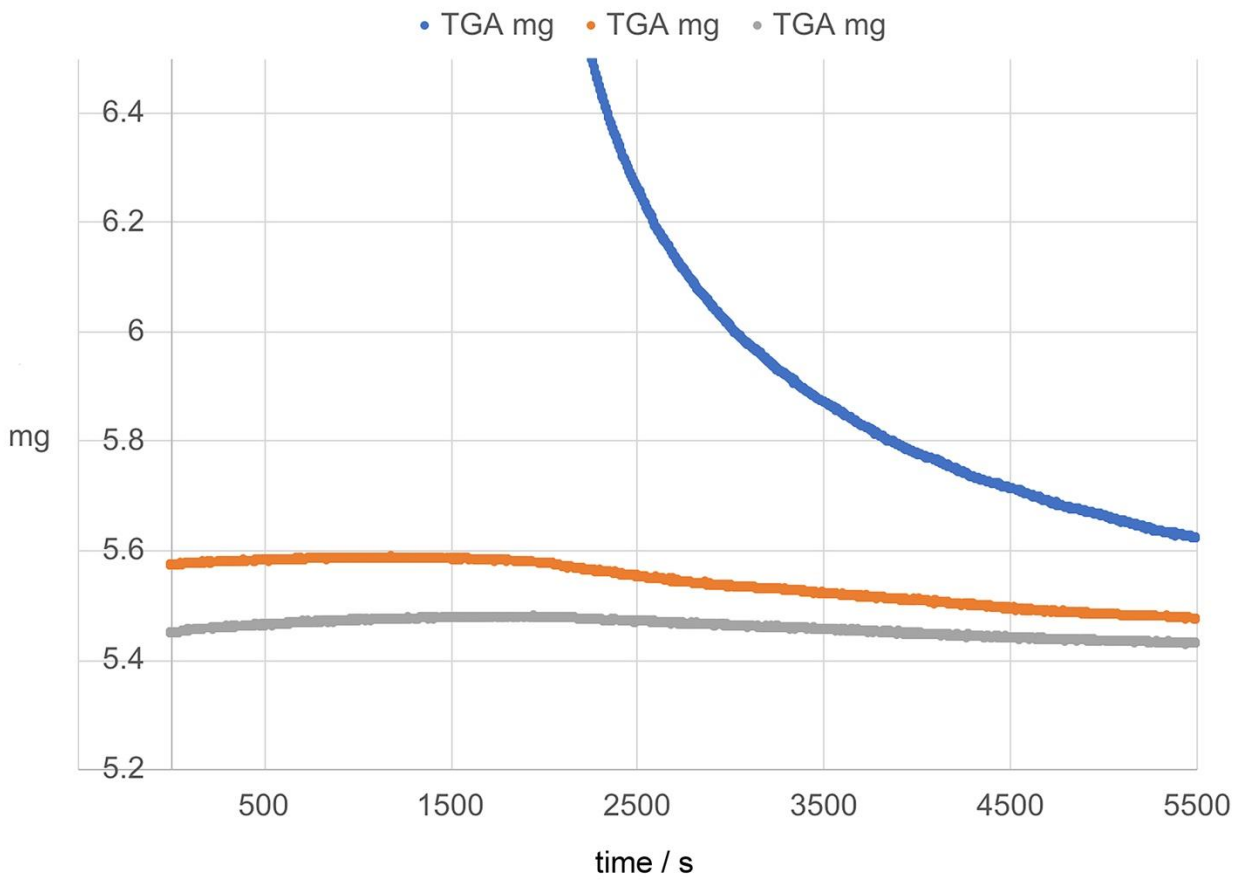


Figure 7: Thermogravimetric Analysis (TGA) of DT-420 on steel. Three consecutive runs with the same coating were performed per the temperature profile shown in Figure 1 (blue: first run; orange: second run; gray: third run). This Figure shows the loss of mass of the DT-420 coating itself after deposition. The slight increase in weight during the initial phases of runs 2 and 3 are due to increased uplift during the heating-up phase. Loss of mass of DT-420 occurs continuously during three consecutive heating cycles.

As shown in Figure 7, the mass of the DT-420 coating decreases continuously during three consecutive heating cycles. The chemical analysis of the emitted vapors (Figure 6) indicates that a mixture of ammonia and ethylene (and potentially other hydrocarbons) are emitted from the coating at 205 °C/400 °F).

Conclusions:

1) DT-420 forms a thermally metastable coating on steel. After the solvent was emitted during the curing reaction, further emission of ammonia and ethylene (and potentially other hydrocarbons) was observed by mass spectroscopy. This indicates a slow chemical transformation of DT-420 to an even more crosslinked structure.
2) No indications for low molecular weight silanes were detected in either run.