COMMITTEE OF EXPERTS ON THE TRANSPORT OF DANGEROUS GOODS AND ON THE GLOBALLY HARMONIZED SYSTEM OF CLASSIFICATION AND LABELLING OF CHEMICALS

Sub-Committee of Experts on the
Transport of Dangerous Goods

Thirty-first session
Geneva, 2-6 July 2007
Item 3 (a) of the provisional agenda

LISTING, CLASSIFICATION AND PACKING

Supplemental information to ST/SG/AC.10/C.3/2007/22
Amendment to UN 3474 for inclusion of 1-HOBt Monohydrate

Transmitted by the expert from the United States of America

1. The purpose of this informal document is to provide additional information in support of ST/SG/AC.10/C.3/2007/22. The test results provided in this document demonstrate that 1-HOBt monohydrate retains its water content in a wide temperature range, 20 ºC to 80 ºC. By comparison, a mixture of anhydrous 1-HOBt and 20 % water will lose its water content at much faster rate in the same temperature range. These results further support classification of 1-HOBt monohydrate as a Division 4.1 desensitized explosive substance to be included in the proper shipping name for UN 3474.

2. Tests were conducted by the Bristol-Myers Squibb Company. The dehydration behavior of several mixtures of 1-HOBT and water were studied. The results are briefly summarized below:

DEHYDRATION BEHAVIOR OF 1-HYDROXYBENZOTRIAZOLE (1-HOBt)

The monohydrate HOBt material showed a weight loss of 11.7% which is close to the theoretical weight loss of 11.8% (Figure 1A). The maximum rate of weight loss occurs between 80-116°C. When the monohydrate material is wetted with 8% water, the weight loss occurs in two distinct steps; the first and second steps have a maximum rate of weight loss between 25-82°C and 82-126°C, respectively (Figure 1B). The temperature range for the maximum rate of weight loss in the second step is similar to that of the monohydrate HOBt material. Therefore, for the monohydrate HOBt material with 8% added water, the first step in the TGA curve largely represents loss of adventitious water (or unbound water) and the second step is loss of the water from the crystalline monohydrate HOBt structure. The rate of water loss for the adventitious water in the first step occurs at a faster rate and lower temperature than the water of crystallization in the second step.

The dehydration behavior for wetted anhydrous HOBt material with 20% water added with and without mixing is shown in curves C and D in Figure 1. It is important to note that while these samples initially contained anhydrous HOBt, conversion to the hydrate form can occur with the addition of water. Laboratory studies have shown that significant conversion of the anhydrous form to the hydrate can occur within 15 minutes of adding stoichiometric amounts of water with mixing. The rate of dehydration will not only depend on the rate and extent of conversion to the monohydrate form but also on the physical properties of the solid such as particle size and degree of crystallinity. In the current study, the two anhydrous samples with 20% water dehydrated at a lower temperature range (25-100 °C) as compared to the HOBt hydrate which dehydrates between 80-116 °C. Dehydration was also completed at lower temperatures; the two wetted anhydrous samples completely dehydrate by 100 °C whereas dehydration is not complete until 116 and 126 °C for HOBt monohydrate and wetted HOBt monohydrate, respectively.

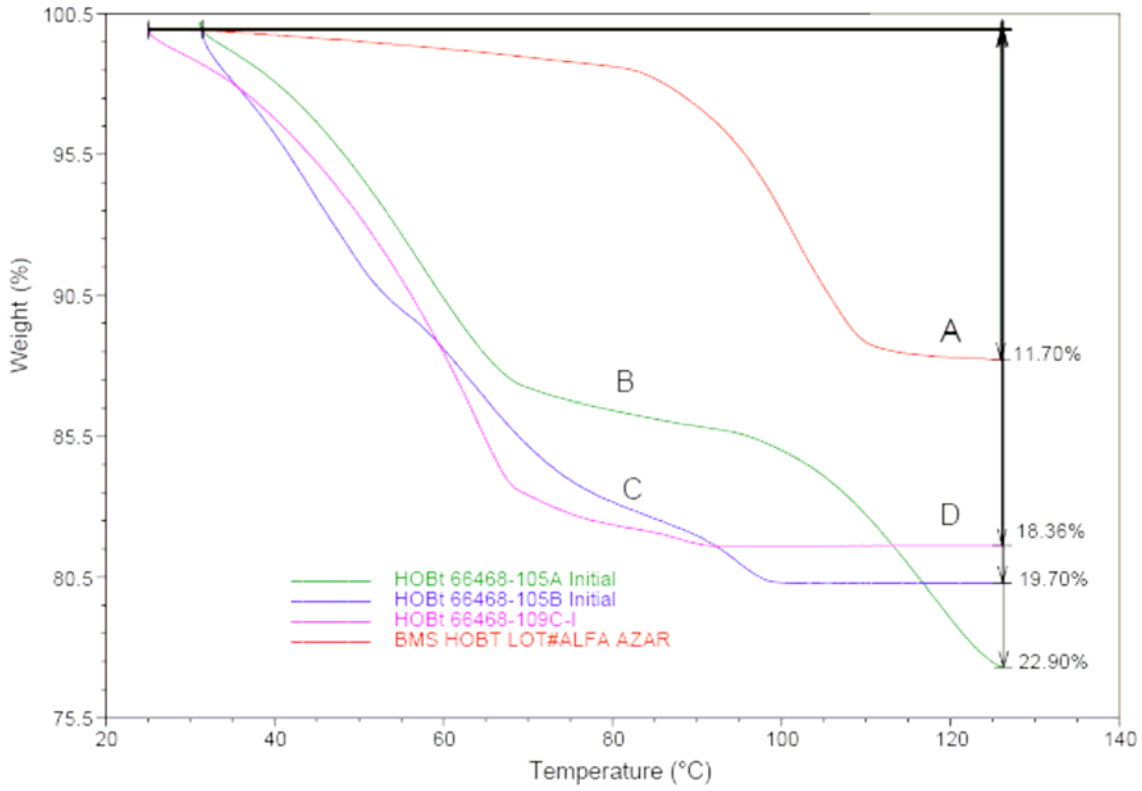

Figure 1: TGA curves for samples which were initially 1-HOBt: (A) Monohydrate form (theoretical weight %: 11.8) as confirmed by GADDS XRD, (B) Monohydrate form mixed with 8% water, (C) Anhydrous Form mixed with 20% water and (D) Anhydrous Form with 20% water unmixed.