



# THE BLACK VAULT

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May 29, 1953

Dear \_\_\_\_\_

As you may well gather from our telephone conversations and my silence during the past two months, the reason you have not heard from me sooner regarding the material (I) which was submitted to us has been that our recent work has not yielded positive results. You may remember that at the time I submitted my first report (August, 1952) we had determined that this material showed spectral characteristics consistent with the presence of  $C\equiv N$ ,  $C\equiv CH$ , or  $SiH$  groups (absorption around  $2200\text{ cm}^{-1}$ ) in addition to the possible presence of carbonyl, amide, and methylene groups.

It was our intention to attempt to determine more about the constituent molecular groupings in the crystalline material by deuteration experiments and you kindly supplied us with some  $CH_3OD$ .

We attempted to deuterate some fragmentary crystals of the substance (I) and on doing so obtained another substance (II). We did not work with the large crystals of (I); but rather with some fragments which may or may not have been identical with the large crystals whose spectral properties have been determined and reported. In any case, the substance (II) showed the presence of deuterium, as evidenced by absorption at  $2630$  and  $2515\text{ cm}^{-1}$  and showed no absorption in the region where there previously had been absorption around  $2200\text{ cm}^{-1}$ . Furthermore, the substance (II) showed no sharp melting point on heating to  $330^\circ\text{ C}$ . Thirdly, on removing the deuterium by boiling with water, the original material was not obtained, since no absorption bands appeared in the substance (III) around  $2200\text{ cm}^{-1}$ .

From the above experiments it may be concluded that either

- substance (I) is a mixture of two materials and we were not using the same substance with which we obtained our original spectral data, or
- substance (I) is decomposed by treating with  $CH_3OD$  and retreating with water to yield a material non-identical with the original compound (I).

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To verify or disprove either of these hypotheses it will be necessary to take one of the larger original crystals and attempt to deuterate that. In view of our lack of success in learning anything definite to date, I have hesitated to do this. If, however, you have no other use for the material, we shall attempt this procedure. As indicated in my report of July 29, 1952, it may be possible thus to learn more about the molecular groupings present in the original crystalline material (I). I await your advice on this matter.

Yours sincerely,  
C

cc: A