

## Preparation of diiodoacetylene, by AXT

**Warning:** Diiodoacetylene is extremely toxic[2] and a shock, heat and friction sensitive explosive[2, 3].

This preparation is a slightly modified version of that of Dehn[1]. 3 g of potassium iodide was dissolved in 40 mL of distilled water in a 100 mL measuring cylinder. A steady stream of acetylene produced by calcium carbide in water was bubbled through the potassium iodide solution. A 12.5% solution of sodium hypochlorite was slowly dripped into the bubbling potassium iodide solution where by the solution turned reddish amber, then pale yellow. The slow addition of hypochlorite was continued until a flocculent white precipitate of diiodoacetylene filled the measuring cylinder and the hypochlorite addition no longer turned the solution yellow (Figure 1).

Figure 1: Colour change, and precipitation of diiodoacetylene



The precipitate was filtered, flushed with cold water, and dried in a cardboard box since diiodoacetylene is light sensitive. It is also volatile and will be lost through sublimation if left uncovered.

The slow addition of the hypochlorite is used to produce the unstable hypoiodite, which reacts with the acetylene to produce diiodoacetylene. The equations for the reactions are as follows:

$$\begin{array}{rcl} CaC_2+2H_2O & \rightarrow & Ca(OH)_2+C_2H_2\\ KI+NaOCl & \rightarrow & KCl+NaOI\\ 2NaOI+C_2H_2 & \rightarrow & 2NaOH+C_2I_2 \end{array}$$

The yield of diiodoacetylene melting at 81°C from this procedure is about 88%. Losses are due to the instability of the hypoiodite which decomposes through the following equations:

 $\begin{array}{rcl} 3NaOI & \rightarrow & 2NaI+NaIO_{3}\\ NaOI+3NaOCl+NaOH+H_{2}O & \rightarrow & 3NaCl+H_{3}Na_{2}IO_{6} \end{array}$ 

## References

- [1] Dehn, W. Journal of the American Chemical Society 34, 1598-1601 (1911).
- [2] Fedoroff, B. et al. <u>Encyclopedia of Explosives and Related Items</u>. vol. 7 pg. H6 & vol. 5 pg. D1298.
- [3] Urben, Peter (editor: Leslie Bretherick). <u>Bretherick's Handbook of Reactive Chemical Hazards</u>. 5th ed. vol. 1, 0985. Butterworth-Heinemann, 1999.