SIMPLE METHOD FOR THE PREPARATION OF ^{36}Cl-LABELLED 2,2-DICHLOROPROPIONIC ACID IN HIGH YIELD

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Dalapon (2,2-dichloropropionic acid) has been considered as one of the original herbicides which is used to control aquatic and non-aquatic plants.[1] Dalapon labelled with ^{36}Cl has been used in studies to determine its effect on marine and non-marine animals and plants.[2,3] Methods have been reported for preparing this compound using isotopic exchange at 120 °C in acetone as exchange medium under vacuum.[4] In the present study this compound has been labelled with labelled sodium chloride at 140 °C and 10^{-2} atm in an evacuated and sealed reaction tube without any other exchange medium. Advantage is taken of the fact that dalapon itself being a liquid with a boiling point of 185-192 °C serves as an ideal isotopic exchange medium. Labelled dalapon was obtained in high yield (90%) after vacuum distillation (65-70 °C, 10^{-2} atm). The radiochemical yield varied between 60 - 70%. TLC-analysis proved a 100% purity of the product.

The preparation of the labelled acid and the salt

In a clean dry reaction tube as shown in Figure (1), 1 - 2 ml of pure dalapon (BASF technical grade, purified by vacuum distillation at 65 - 70 °C and 10^{-2} atm) was added to 5 - 50 mg of radioactive $Na^{36}Cl$ (Amersham, specific activity 3 mCi/1 mg ^{36}Cl). The reaction tube

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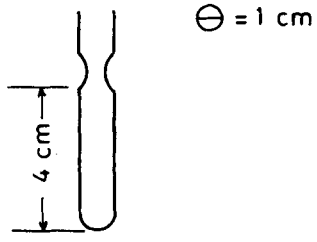
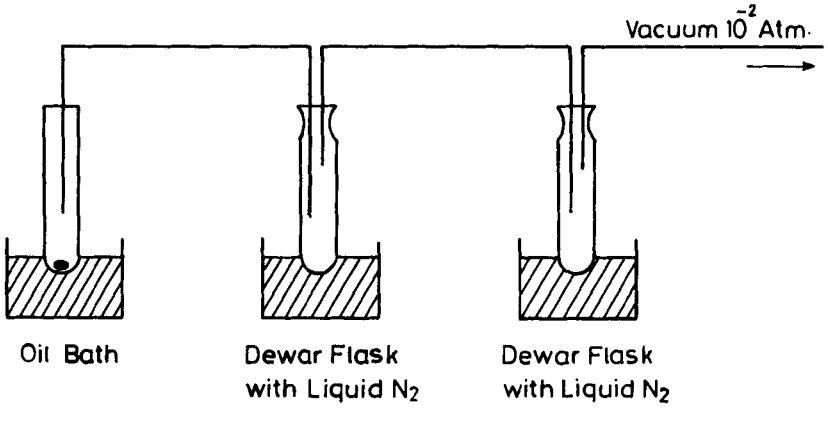


FIG·(1) REACTION TUBE FOR LABELLING DALAPON WITH $Na^{36}Cl$



FIG·(2) ARRANGEMENT OF DISTILLATION – APPARATUS FOR SEPARATION OF LABELLED DALAPON

was then evacuated with a water jet pump and sealed. The reaction mixture was distilled at 65 - 70 °C and 10^{-2} atm (see Figure (2)). The pure labelled dalapon was collected in a clean dry receiver.

The chemical and radiochemical purity were determined by applying the sample in a volume of 5 µl of freshly distilled labelled dalapon on TLC-plate (silicagel, 200 x 0.25 x 1 mm, Merck AG, Federal Republic of Germany) and allowed to dry. On the same plate one drop (5 µl) of non-radioactive dalapon with known purity was placed and allowed to dry. The chromatogram was developed by chromatography, using a mixture of 10 volumes of acetic acid and 190 volume of chloroform.[5] The plate was allowed to dry and the position of the inactive dalapon determined by spraying of the plate with bromocresol solution.[5] The distribution of radioactivity was determined by scanning the plate using II-Thinlayer Chromatography Scanner (Berthold, Federal Republic of Germany). We found that the R_F value for the inactive dalapon band is identical with the value for the radioactive dalapon band. The salt was prepared by adding 0.008 M of Na_2CO_3 to 20 ml of tetrachloroethylene. Then 0.013 M of labelled dalapon were added to the mixture drop by drop and the mixture was heated at 50 °C for one hour.[6] The product was isolated by filtration on glas filter disc (G-2) and purified by washing with tetrachloroethylene, acetone and ether solvents. Dry dalapon salt was then collected.

Discussion

The method reported here for the preparation of labelled dalapon is mainly dependent on the solubility of $Na^{36}Cl$ in dalapon under the above mentioned conditions. This method has the advantage over the previously reported method[4] in that the reaction procedure is safe avoiding the danger of explosion which might occur with acetone. In addition, due to the absence of acetone in our method the separation of dalapon from the reaction mixture is very easy. Our chromatographic analysis indicated that the distillation product contained only one compound which is the dalapon. Few experiments were performed to evaluate the effect of temperature and the reaction time on the exchange reaction. Poor yields were obtained at 80 - 120 ^{o}C. At 150 - 180 ^{o}C the product undergoes decomposition. The exchange for 2, 4, 8 hours and overnight was investigated. Low specific activities were found with less than 24 hr. The choice of 140 ^{o}C as the optimum reaction temperature and the 24 hr as the optimum reaction time are due to the high yield of labelled dalapon. The labelling yield was found also to be dependent on the volume of the reaction tube. It was observed that the 3 ccm capacity reaction tube offers 3 - 4 times higher yields than the 10 ccm tube.

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