Automated Syntheses of Carrier-free 77Br-labelled Alkylbromides

VI. 2 Automated Syntheses of Carrier-free ^{77}Br-labelled Alkylbromides

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The automated synthetic method developed for carrier-free ^{18}F-labelled alkylfluorides is available to preparation of similar organic-compounds labelled with other radiohalogens. In the case of ^{77}Br-labelling, it is expected that the automated synthesis can be performed by nucleophilic displacement with the corresponding iodo-organics and/or interhalogen exchange reaction with the corresponding bromo-organics.

In the present studies, we have tried to establish the above approaches by using carrier-free ^{77}Br source, fixed on silver-wool, in a similar manner as mentioned in the previous report (VII).

Carrier-free ^{77}Br was produced by bombarding arsenic pentoxide target with 30 MeV alpha-particles in the external beam. The target was prepared by pressing about 1 g of arsenic pentoxide powder onto a grooved alminum plate and covering it with a 0.03 mm titanium foil. The target was then clamped into the target holder. Arsenic pentoxide has been chosen to be the most convenient material as the target since it is readily dissolved in water after bombardment. During bombardment, the beam current was normally restricted to 5 μA, while the back of target plate and the retaining foil were cooled by water and an air jet, respectively. After bombardment, the target was left for at least 12 hrs to allow the decay of short lived contaminants, notably ^{18}F from the ^{16}O(α,pn)^{18}F reaction.

Chemical processing of carrier-free ^{77}Br was carried out as follows. The titanium cover was removed and the arsenic pentoxide was washed off as a slurry from the grooved target plate with 10 ml of water. The slurry was then transferred to a distillation flask with further 5 ml of water. After the distillation apparatus which is consisted of distillation flask, hydrogen bromide generator, silver-wool column and 0.1 N sodium hydroxide trap cooled in an ice bath was completely assembled, about 30 ml of conc.sulfuric acid and 5 g of potassium permanganate were added to the flask via its side arm. The stopper was replaced in the side arm and the apparatus was flushed through nitrogen carrier-gas at a flow rate of 50 ml min^{-1}. The distillation flask was then heated gently to about 150°C. Carrier-free ^{77}Br distilled was converted to H^{77}Br by passing through the hydrogen bromide generator (red phosphorus column) and collected by the successive silver-wool column. The silver-wool used was freshly prepared for each distillation by the electrolytic oxidation in sodium solution. After distillation has finished, the generator and the wall of the apparatus before the silver-wool column were heated by using a heat-blower in order to release the rest of H^{77}Br. By this procedure,

almost all of $H^{77}Br$ was completely caught on the top of silver-wool column. The silver-wool column was then removed from the apparatus and dried by blowing dry helium gas at 110°C.

Automated syntheses of ^{77}Br-labelled alkylbromides by nucleophilic displacement and interhalogen exchange reaction were carried out in similar manner as mentioned in the previous report.

Carrier-free and non-carrier-free ^{77}Br labelled alkylbromides prepared are shown in Table 1 and 2, respectively.

Table 1. Carrier-free ^{77}Br-labelled alkylbromides prepared by nucleophilic displacement.

Reactant	Product (Carrier-free)	Relative yield
CH_3I	$CH_3{}^{77}Br$	156
C_2H_5I	$C_2H_5{}^{77}Br$	110
$n\text{-}C_3H_7I$	$n\text{-}C_3H_7{}^{77}Br$	100
$i\text{-}C_3H_7I$	$i\text{-}C_3H_7{}^{77}Br$	109
$n\text{-}C_4H_9I$	$n\text{-}C_4H_9{}^{77}Br$	98
$i\text{-}C_4H_9I$	$i\text{-}C_4H_9{}^{77}Br$	26
CH_2CHCH_2I	$CH_2CHCH_2{}^{77}Br$	33

Table 2. Non-carrier-free ^{77}Br labelled alkylbromides by interhalogen exchange reaction.

Reactant	Product	Relative yield
C_2H_5Br	$C_2H_5{}^{77}Br$	100
$n\text{-}C_3H_7Br$	$n\text{-}C_3H_7{}^{77}Br$	71
$i\text{-}C_3H_7Br$	$i\text{-}C_3H_7{}^{77}Br$	304
$n\text{-}C_4H_9Br$	$n\text{-}C_4H_9{}^{77}Br$	167
$i\text{-}C_4H_9Br$	$i\text{-}C_4H_9{}^{77}Br$	44
$s\text{-}C_4H_9Br$	$s\text{-}C_4H_9{}^{77}Br$	222
$t\text{-}C_4H_9Br$	$t\text{-}C_4H_9{}^{77}Br$	51
CH_2CHCH_2Br	$CH_2CHCH_2{}^{77}Br$	-
CH_2Br_2	$CH_2Br{}^{77}Br$	-
CH_2BrCl	$CH_2{}^{77}BrCl$	-