

## Short Research Article

# A fast and simple method of isomerization for the preparation of ammonium ( $^{14}\text{C}$ )thiocyanate from ( $^{14}\text{C}$ )thiourea<sup>†</sup>

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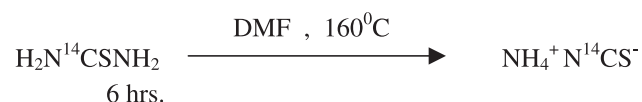
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## Introduction

Ammonium thiocyanate labelled with carbon-14 is used as an alternative to tritiated water for tracing of water-flow in water-driven oil production.<sup>1,2</sup> We have prepared ammonium [ $^{14}\text{C}$ ]thiocyanate from potassium [ $^{14}\text{C}$ ]cyanide and [ $^{14}\text{C}$ ]thiourea.<sup>3,4</sup> We now report a simple and rapid isomerization method for the preparation of ammonium [ $^{14}\text{C}$ ]thiocyanate from [ $^{14}\text{C}$ ]thiourea.

The reaction scheme is shown below.



[ $^{14}\text{C}$ ]Thiourea was isomerized to ammonium [ $^{14}\text{C}$ ]thiocyanate by heating a DMF solution (11.5% w/v) at 160°C for 6 h. The product formed was purified by silica-gel column chromatography. A radiochemical yield of 88% was obtained based on [ $^{14}\text{C}$ ]thiourea on the 1.5 mmol scale. The specific activity of the product obtained was 52.25 mCi/mmol (1.94 GBq/mmol) and the radiochemical purity was greater than 99%.

## Results and discussion

The isomerization of thiourea to ammonium thiocyanate is a convenient and safe method for the preparation

of ammonium [ $^{14}\text{C}$ ]thiocyanate. The isomerization reaction carried out in aqueous solution requires 24 h heating in sealed conditions for optimum radiochemical yield. We tried different aprotic solvents for the isomerization reaction of thiourea to ammonium thiocyanate and DMF was found suitable. In DMF we obtained our optimal yield without the need for sealed conditions.

The chemical yield of isomerization reaction when carried out using 1.0 ml of DMF in round-bottomed flask at 80°C–120°C for 2 h was <10%, whereas a yield of 85.97% was obtained when heating was carried out at 160°C for 6 h. By using different concentrations of thiourea in DMF at 160°C for 6 h we obtained our highest yield of 87.5% when the concentration of thiourea in DMF was 10.13%. Table 1 shows that refluxing a 7–15% solution of thiourea in DMF at 160°C for 6 h were suitable high-yielding conditions for the reaction.

The optimum reaction conditions chosen from the above were used for the synthesis of ammonium [ $^{14}\text{C}$ ]thiocyanate. The results obtained from a set of three experiments are shown in Table 2. The method requires less time and no sealed conditions. The procedure described here represents a simple and fast

**Table 1** Isomerization of thiourea to ammonium thiocyanate using five different concentrations in DMF (160°C for 6 h)

Concentration of thiourea in DMF (%)	5	7.6	10.13	15.2	30.14
Chemical yield of ammonium thiocyanate(%)	75.1	86.2	87.5	85.9	79.5

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**Table 2** Radiosynthesis of ammonium [ $^{14}\text{C}$ ]thiocyanate

$^{14}\text{C}$ Thiourea		DMF ( $\mu\text{l}$ )	Ammonium [ $^{14}\text{C}$ ]thiocyanate		Radiochemical yield (%)
mmol	Radioactivity (mCi)		mmol	Radioactivity (mCi)	
1.48	78.4	1000	1.33	68.7	87.6
0.64	34.8	500	0.59	31.2	89.7
1.52	81.7	1000	1.38	72.1	88.3

method of isomerization for the preparation of ammonium [ $^{14}\text{C}$ ]thiocyanate from [ $^{14}\text{C}$ ]thiourea.

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