donner l'espèce SO_x. conduisant au sulfite en milieu réducteur, une autre partie subsiste à l'état d'ions S^+ qui sont oxydés en sulfate au moment de la dissolution.

L'analyse des états chimiques finaux permet de déterminer l'ordre de grandeur de chacun des états intermédiaires:

– à l'état solide

S^{2-}	10%
S^0	55%
S^+	15%
SO_x	20%

– en solution aqueuse

S^{2-}	12%
S^0	60%
S^+	18%
SO_x	10%

Nous remercions vivement le personnel du Service de la Pile Universitaire de Strasbourg pour les nombreuses irradiations nécessaires pour l'accomplissement de cette étude, ainsi que Monsieur HEITZ pour l'intérêt qu'il a bien voulu porter à ce travail et Monsieur BONNENFANT pour son aide efficace.

SHORT COMMUNICATIONS

Preparation of Sodium O. Iodohippurate ^{131}I

By GLADI N. B. DE SALAS and ALDO E. A. MITTA, Comisión Nacional de Energía Atómica, Departamento de Química, División Moléculas Marcadas, Buenos Aires, Argentina

(Received December 1, 1969, in amended form April 28, 1970)

Introduction

Several methods are known for the preparation of labelled sodium o-iodohippurate-^{131}I, some by means of $Na^{131}I$ [1, 2, 3, 4, 6, 7, 8] and others by means of $Cl^{131}I$ [5].

We have investigated the method of VEALL et al. [2], and taken into account the influence of some variables such as p_H, light, temperature and mass of both reagents and oxidative agents. If our prescription is followed, radio-iodohippurate is obtained by a rapid and simple method with a radiochemical yield of 98–100%.

Labelling Technique

An aqueous solution of inactive sodium-iodohippurate (1.5 ml, 240 mg/ml) is placed in a glass stoppered test tube of 5 ml capacity. A few drops (3–20 mCi) of carrier- and reductant-free $Na^{131}I$ are added ($\leqslant 50$ mCi/ml), as well as a drop of 0.2% KIO_3. The p_H is adjusted to 5.6 with 0.5 N HCl and the tube is stoppered and heated for 90 min in a water bath under the light of a 150 watt lamp placed at a distance of 15 cm from the reaction tube. After the heating a drop of 0.1 M $Na_2S_2O_3$ is added, the p_H is adjusted to 8 and the solution is sterilized for 30 min in an autoclave. The radiochemical yield is about 98–100%.

Influence of Variables

We have studied the influence of reaction time, p_H and nature of oxidant to arrive at the most suitable labelling method, which is the one described above.

For this purpose the activity of inorganic radioiodide in our sample was determined by descending paper chromatography [9] (butylalcohol, acetic acid and water, 4:1:1, Whatmann 3 MM paper, developping time 90 min. R_f radio-iodohippurate = 0.85, R_f iodide = 0.10), by paper electrophoresis [9] (Na acetate – acetic acid buffer, p_H 5.5, 0.0075 molar, Whatmann 3 MM paper, 300 V, 90 min, iodide migrates 10 cm, radio-iodohippurate does not migrate) and thin layer chromatography (silicagel G, 1 molar HCl, developping time 15 min, R_f radio-iodohippurate = 0.29 ± 0.01, R_f iodide = 0.8).

It was noticed that in the absence of light the yield of radio-iodohippurate decreases appreciably [4]. The

1. M. TUBIS, E. POSNIK and R. A. NORDYK, Proc. Soc. Biol. Med. **103**, 497 (1961).
2. A. E. A. MITTA, A. FRAGA and N. VEALL, Int. J. Appl. Rad. Isotopes **12**, 146 (1961).
3. K. E. SCHEER and W. MEIER-BORST, Nuclear Med. **2**, 193 (1961).
4. K. HORIUCHI and H. BEHRENS, Primera Conferencia Interamericana de Radioquímica, Montevideo, 1963 (Union Panamericana, Washington 1965) 273.
5. L. J. ANGHILIERI, Int. J. Appl. Rad. Isotopes **15**, 95 (1963).
6. I. ELEKES and D. KORITSANSZKY, Gyogyszereszet 10 No. 2, 65 (1966), Nuclear Sci. Abstr. **20**, 43388 (1966).
7. G. C. JOSHI, B. SINGH, D. N. ZUTSHI and K. P. CHAKRABORTY, Proceedings of the Nuclear and Radiation Chemistry Symposium, 1966 (Bombay, Dept. of Atomic Energy, 1966) 303.
8. R. S. MANI and R. J. P. PRABAKARAN, Current Sci. **35**, (1), 16 (1966).
9. G. MAGNUSSON, Nature **195**, 591 (1962).

same is true if the reaction time is reduced, the p_H is too low or too high, the iodohippurate concentration too low or if H_2O_2 or chloramine T are used as an oxidant instead of KIO_3.

The data obtained in our experiments are shown in the tables.

Table 1. *Influence of p_H and reaction time on the yield of radio-iodohippurate*

p_H	Iodohippurate-^{131}I yield (percent) 30 min	90 min
4.4 ± 0.1	4	13
4.7 ± 0.1	37	63
4.9 ± 0.1	42	89
5.1 ± 0.1	52	93
5.3 ± 0.1	72	98
5.6 ± 0.1	72	99
5.7 ± 0.1	69	99
5.9 ± 0.1	63	98
6.2 ± 0.1	54	86
6.7 ± 0.1	45	72
7.8 ± 0.1	9	21
10.4 ± 0.1	0	0

The oxidant is KIO_3, the concentration of iodohippurate 240 mg/ml.

Table 2. *Influence of the iodohippurate concentration on the yield of radio-iodohippurate*

Concentration of Iodohippurate mg/ml	Iodohippurate-^{131}I yield (percent)
0.25	17
2.5	31
126	90
210	99
252	99
360	91

The oxidant is KIO_3, the $p_H = 5.6$, the reaction time 90 min.

Table 3. *Influence of reaction time and the nature of the oxidant on the yield of radio-iodohippurate*

Reaction time (min)	Iodohippurate-^{131}I yield (percent) KIO_3	Chloramine T	H_2O_2
15	57	53	56
30	66	55	69
60	94	63	80
90	99	70	86
120	99	79	89
180	100	87	89
210	99	87	91

The concentration of iodohippurate is 240 mg/ml, the $p_H = 5.6$.

On the Isotopic Exchange Reaction in Solid State between Cr(III) Oxinate and Dopant $^{51}Cr^{3+}$ Ions

By E. LAZZARINI, T. ANNONI and A. L. FANTOLA-LAZZARINI, CESNEF, Politecnico di Milano, Via Ponzio 34/3, 20133 Milano

With 3 figures. (Received May 20, 1970)

While the role of chemical intermediates in thermal annealing of Szilard-Chalmers recoils was claimed and proved effective in many cases by several authors, on the contrary, less direct information is available on this topic for the isotopic exchange reactions in solid state [1, 2]. This present communication deals with the above mentioned aspect of the isotopic exchange reactions in solid state, as it was observed in the system $^{51}Cr^{3+}$–Cr(III) oxinate. Chromium oxinate was prepared according to the method described by A. ABLOV [3] and purified by repeated crystallizations from chloroform. 5 g of the compound were suspended in 100 ml of benzene and 2 mCi of $^{51}CrCl_3$, dissolved in about 1 ml of 1 N HCl, were added to the suspension. The specific activity of $CrCl_3$, as given by the supplier, was 80 Ci/g. The suspension was dried in a rotating evaporator at room temperature; finally the Cr(III) oxinate crystals doped with ^{51}Cr were collected from the flask and dried for a further twelve hours at 10 μHg. The estimated concentration of dopant Cr^{3+} was $\sim 5 \times 10^{-5}$ g ion/complex mole.

Portions of about 10 mg of doped crystals were sealed in Pyrex ampoules in air atmosphere or in vacuum (1 μHg) and then heated in thermostatic baths regulated within ± 1 °C.

Heatings were carried out at the following temperatures: 66, 80, 110, 130, 155, 161, 181 and 205 °C. After heating, the samples were dissolved in methyl alcohol, containing Cr^{3+} carrier, and the solutions were analyzed by means of thin layer chromatography (TLC) on Eastman chromatogram sheets K 301 V. The solvent consisted in a mixture of benzene and methyl alcohol (4:6 v/v). The chromatogram was covered after the development with a self-adesive tape and cut into strips of 1 cm along the migration direction. The ^{51}Cr activity of each strip was counted by means of a 2″×2″ NaI(Tl) well-type crystal. Integral counts were taken. A typical histogram of TLC

1. I. GAINAR, Rev. Roumaine Phys. **13**, 645 (1968).
2. S. KHORANA and D. R. WILES, J. Inorg. Nucl. Chem. **31**, 3387 (1969).
3. A. ABLOV, Bull. Soc. Chim. France **53**, 234 (1933).