

## Chemistry of $^{128}\text{I}$ in Alkaline Aqueous Solutions of Sodium Iodate Activated by Thermal Neutrons

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The retention of  $^{128}\text{I}$  in crystalline sodium iodate at room temperature irradiation is found to be 67.8%, which is quite different from the value of 56.0% observed on irradiation at liquid-nitrogen temperature. Radiative-neutron capture in frozen alkaline aqueous solution of the target irradiated at liquid-nitrogen temperature yields a retention value of 35.1% compared with a value of 23.9% at room temperature for the same solution. Dilution of alkaline aqueous solution with additives (*i.e.* KCl, NaCl, KOAc and NaOAc) prior to neutron irradiation in varying amounts results in an increase of retention in general; also the yield of radioperiodate increases almost reaching a plateau at a molar ratio of additive to target of *ca.* 1:1. The results are explained in the light of a model which invokes the oxidizing-reducing nature of the intermediates produced during neutron irradiation.

The chemical effects following thermal-neutron capture in inorganic systems have been extensively studied for several years, but most of these investigations have concerned the behaviour of hot halogen atoms in crystalline-phase irradiation.<sup>1</sup> The results of such studies show clearly that the ultimate chemical fate of atoms undergoing nuclear transformation in halogen oxyanions is determined by complex interactions of nuclear, thermal, radiation and composition effects. Earlier reports on thermal-annealing experiments in  $\text{NaIO}_3$  show that the retention of recoil  $^{128}\text{I}$  increases on heating the irradiated solid.<sup>2-5</sup> Furthermore, the occurrence of thermal annealing reactions at room temperature and even to  $-80^\circ\text{C}$  has been reported in solid iodate targets.<sup>6, 7</sup> So far retention in iodates and periodates in the solution phase has not received much attention, although the hot-atom chemistry of such systems appears an obvious extension of investigations in this direction. The study of hot-atom reactions in the solution phase also seem to offer certain prospects for gaining a more thorough insight into the mechanism of processes taking place in the radical nest.

Results of  $(n, \gamma)$  reactions for iodates and periodates in aqueous solution are few in comparison with those in crystalline solids.<sup>8-10</sup> Cleary *et al.*<sup>2</sup> first showed that the radioiodate fraction produced by neutron irradiation of a neutral aqueous solution of iodate varies with the addition of oxidizing and reducing agents. They tried to explain their results on the basis of a ligand-loss hypothesis. Also, other studies on the  $(n, \gamma)$  irradiation of neutral iodate systems present a lower limit of retention that can be achieved in the neutral aqueous phase by the choice of a suitable reducing agent, and their explanation for the observed retention and yields are far from satisfactory.<sup>2, 4</sup> Arnika *et al.*<sup>8, 9</sup> found that the retention from  $\text{LiIO}_3$  and  $\text{HIO}_3$  in aqueous solution increases with concentration. However, they were unable to find any radioperiodate in the targets studied. Recently some results for iodates and periodates

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have been published by the present authors.<sup>11–13</sup> This work was undertaken as a reinvestigation of the fate of recoil iodine-128 atoms in an  $\text{NaIO}_3$  target, in both the solid and solution phases (particularly in alkaline aqueous media), and to examine the influence of additives, *i.e.* KCl, NaCl, KOAc and NaOAc, with a view to comparing the effect of various chemical and physical processes on the observed data of recoil- $^{128}\text{I}$  stabilization.

## EXPERIMENTAL

### THERMAL-NEUTRON IRRADIATION OF SOLIDS

300 mg of crystalline AnalaR  $\text{NaIO}_3$  was irradiated by thermal neutrons from a 300 mCi (Ra–Be) neutron source having an integrated flux of  $3.2 \times 10^6$  neutron  $\text{cm}^{-2} \text{s}^{-1}$ . The neutron source was surrounded by a cylindrical block of paraffin for thermalizing fast neutrons. The concomitant  $\gamma$ -dose associated with the neutron source was *ca.* 172 rad  $\text{h}^{-1}$  as estimated by Frick dosimetry.

### THERMAL-NEUTRON IRRADIATION OF SOLUTIONS

Solutions of  $\text{NaIO}_3$  ( $0.076 \text{ mol dm}^{-3}$ ) were made by dissolving 0.300 g of target material in  $20.0 \text{ cm}^3$  of  $0.1 \text{ mol dm}^{-3}$  NaOH solution at a pH of *ca.* 12.0. For the irradiation of these solutions a hollow cylindrical paraffin block of *ca.* 3 cm thickness was made inside a beaker. The neutron source was placed in the inner hollow portion within the beaker. The beaker and neutron source were then surrounded by a second beaker of capacity  $400 \text{ cm}^3$ . Under this experimental set-up thermal-neutral irradiation of solutions of  $\text{NaIO}_3$  along with the additives was performed by keeping the solution in the second beaker.

### THERMAL-NEUTRON IRRADIATION OF FROZEN ALKALINE AQUEOUS SOLUTION

The alkaline aqueous solutions of the target were frozen drop by drop in a soda-glass test tube kept at liquid-nitrogen temperature and then dipped in liquid nitrogen contained in a Dewar together with the neutron source.

The three stable radioactive products, *i.e.* iodide, iodate and periodate, were separated by fractional-precipitation and solvent-extraction methods.<sup>14</sup> The activity was corrected for time-decay and density following the method of Aten.<sup>15</sup> Throughout the experiment irradiations was performed for 3 h.

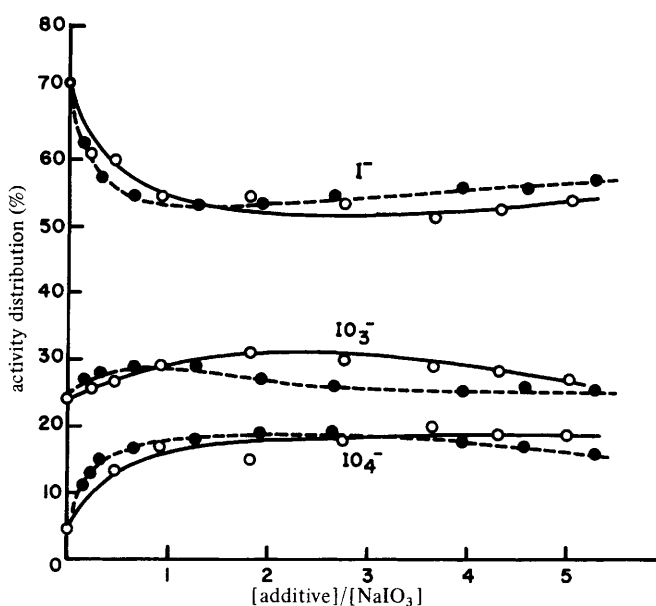
## RESULTS AND DISCUSSION

Retention and yields expressed in % for anhydrous sodium iodate in the crystalline phase and in alkaline aqueous solution for room-temperature and liquid-nitrogen-temperature irradiation are given in table 1. Results from this work and from earlier investigations have revealed that thermal-neutron capture in crystalline  $\text{NaIO}_3$  at room temperature produces a significantly different pattern of chemical consequences from those observed in chlorates and bromates. For example, a much larger fraction of radioactive halogen is retained by the target  $\text{IO}_3^-$  ion than  $\text{ClO}_3^-$  and  $\text{BrO}_3^-$  ions, where retentions in the range *ca.* 5–30%<sup>16–21</sup> have been observed for similar irradiations. The present retention values, 67.8 and 35.0%, for the crystalline solid and for the frozen solution are in good agreement with the results of Cleary *et al.* (67.0%)<sup>2</sup> and Ambe and Saito ( $36.0 \pm 1\%$ ),<sup>4</sup> respectively, which confirms the accuracy and reproducibility of the present results. The values of retention and yields reported are the average of several independent determinations with a reproducibility of  $\pm 1\%$ .

The (n,  $\gamma$ ) reaction was brought about in the  $\text{NaIO}_3$  target in the presence of the additives KCl, NaCl, KOAc and NaOAc in the solution phase prior to neutron irradiation of an alkaline aqueous solution of the sodium iodate, and their consequent effects on the retention and yields during irradiation are presented in fig. 1 and 2. The

**Table 1.** Distribution of recoil  $^{128}\text{I}$  activity in forms of iodide, iodate and periodate ions produced during thermal-neutron irradiation of  $\text{NaIO}_3$  (target  $\text{NaIO}_3$ )

state of sample during irradiation	$T/^\circ\text{C}$	concentration / $\text{mol dm}^{-3}$	activity distribution (%)		
			$\text{IO}_3^-$	$\text{I}^-$	$\text{IO}_4^-$
crystalline solid	25.0	—	67.8	27.9	4.3
crystalline solid	-196.0	—	56.0	42.0	2.0
frozen alkaline aqueous solution	-196.0	0.076	35.1	56.7	8.2
alkaline aqueous solution	25	0.076	23.9	71.7	4.4

**Fig. 1.** Distribution of recoil iodine-128 activity in the form of  $\text{I}^-$ ,  $\text{IO}_3^-$  and  $\text{IO}_4^-$  ions produced during thermal-neutron irradiation of  $\text{NaIO}_3$  in alkaline aqueous media containing (—○—)  $\text{NaCl}$  and (---●---)  $\text{NaOAc}$  additives.

major observations are summarized below. (i) The retention and yields found in the alkaline aqueous phase are quite different from those observed in the crystalline solid. (ii) The retention at room temperature is 67.8%, while the retention measured by thermal-neutron irradiation of an alkaline aqueous solution was found to be 24.0%. Also, the retention in the crystalline phase under room-temperature irradiation is higher than the value observed at liquid-nitrogen temperatures. (iii) A significant yield of radioperiodate from alkaline aqueous solutions has been observed. (iv) The activity distribution depends on the nature of the additives. On increasing the concentration of an additive, there is a sharp increase in retention of 4–7%, as is seen in fig. 1 and 2. The yield of radioiodide decreases from 72% to ca. 52%, while the corresponding yield of radioperiodate increases from 4% to ca. 18%.

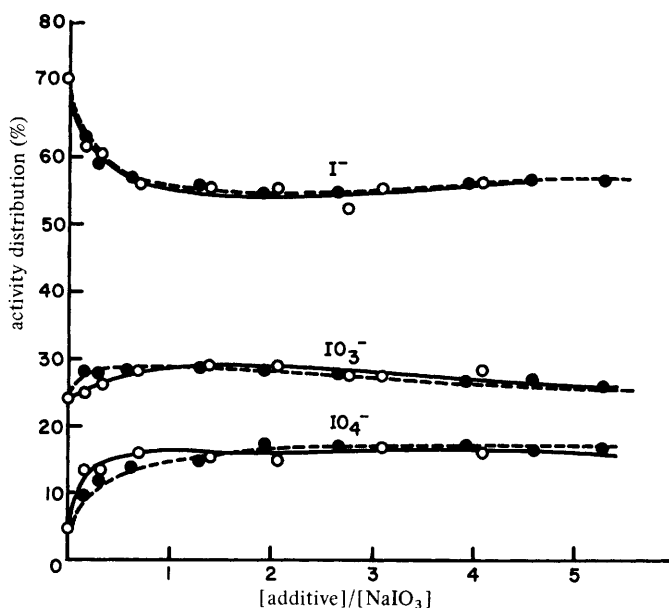
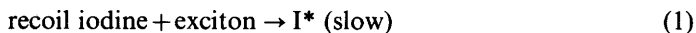


Fig. 2. Distribution of recoil iodine-128 activity in the form of  $\text{I}^-$ ,  $\text{IO}_3^-$  and  $\text{IO}_4^-$  ions produced during thermal-neutron irradiation of  $\text{NaIO}_3$  in alkaline aqueous media containing (—○—) KCl and (—●—) KOAc additives.

In a separate set of experiments it was observed that on changing the concentration of targets and additives while keeping the concentration ratio the same, no appreciable change in retention and yields was observed. In a series of parallel studies, the neutron-irradiated solid  $\text{NaIO}_3$  was dissolved in an aqueous solution containing additives of the same concentrations as were used in the case of the alkaline aqueous phase; again no change in retention and yields was observed.

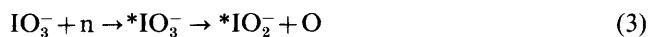
The present experiment and those of Cleary *et al.*<sup>2</sup> indicate that a large fraction of  $^{128}\text{I}$  atoms is initially in the zero oxidate state, except in the case of internal conversion. Therefore, the first reaction the  $^{128}\text{I}$  atom undergoes after recoil is the insertion of  $\text{H}_2\text{O}$ , forming  $^{128}\text{I}^-$  or  $^{128}\text{IO}^-$ . Various experiments show that the radioiodate and radioperiodate are influenced by the additives.<sup>10–12</sup> In the present case, for  $\text{NaIO}_3$  the retentions and yields of iodate and periodate are increased while those of radioiodide decrease, in line with the findings of Bera and Shukla<sup>10</sup> for a  $\text{KIO}_3$  target. This is probably due to a stepwise oxidation reaction by oxidizing species formed either from local radiolysis or from additives, as discussed later.

The effect of the duration of the neutron irradiation on the value of the retention can be accounted for by either of two extreme phenomena.<sup>22</sup> First it is assumed that the initial reaction of the recoil atom is with an electron or hole trapped the  $\gamma$ -ray interaction which produces it. Thus

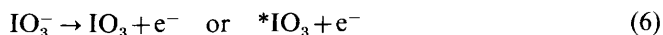


Also, upon exposure of crystalline iodates to energetic radiation, the  $\text{IO}_3^-$  ion undergoes excitation and ionization leading to rupture of the iodine–oxygen bonds.

A number of reactions can be envisaged, but many of these are excluded by considerations of stability, electron affinity *etc.* of the fragments. The primary reactions which are most important in the room-temperature irradiation of iodates are the following:



The existence of the above species in the iodates has been assumed, but not confirmed. However, the existence of  $\text{Br}^-$ ,  $\text{BrO}^-$ ,  $\text{BrO}_2^-$  *etc.* in bromates, in both solid and solution phases, has been confirmed by chemical analysis.<sup>23-26</sup> In cases where free radicals are formed in the primary processes,



these will capture free electrons, forming  ${}^*\text{IO}_3^-$ .<sup>27</sup> Electrons may also be trapped at anion vacancies, forming  $\text{IO}_3^{2-}$  as



The reactive intermediate  $\text{IO}_3^{2-}$  formed may undergo dehydration to yield  $\text{IO}_2$  and  $\text{OH}^-$ :



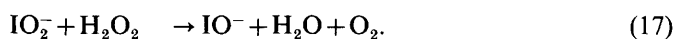
Also, the oxygen atom will remain trapped in the neighbourhood of the sister fragment and react with  $\text{IO}_3^-$ , forming  $\text{IO}_4^-$ . The formation of  $\text{IO}_4^-$  may also occur as follows:



In the radiolysis of pure aqueous solutions of iodates it has been demonstrated<sup>28, 29</sup> that various transients like  $\text{IO}$ ,  $\text{IO}_2$  and  $\text{IO}_3$  are formed owing to the interaction of primary radiolytic species, *viz.*  $\text{e}_{\text{aq}}^-$ ,  $\text{H}$ ,  $\text{OH}$  *etc.* The following sequence of reactions is envisaged:



These reactions explain the degradation of iodate ions *via* various steps. The formation of various radioiodine fragments in higher oxidation states also depends upon the availability of  $\text{OH}$  radicals, as expected from both local radiolysis and internal conversion. Some  $\text{H}_2\text{O}_2$  may also be formed, and according to Haissinsky *et al.*<sup>30</sup>  $\text{H}_2\text{O}_2$  ( $\text{OH} + \text{OH}$ ), if produced, will reduce the intermediate radioiodite ion as



In comparison with the solid-state case, the probability of recombination is much less pronounced in solution because of the possibility of a redox reaction occurring in the aqueous phase. Therefore the factors that account for the thermalization of recoil atoms may be considered as follows: (i) with an increase in the concentrations of the additives, the ion sphere gradually decreases, (ii) with an increase in the masses of the additives, fewer collisions are required for the thermalization of recoil atoms, (iii) the water molecules in the second, third and higher hydration spheres surrounding a target ion are replaced by the additives and in addition water molecules occupy a smaller volume, (iv) the structure of the solution orients towards a close-packed one and finally (v) the mean distance between the target ions may form a hot reaction zone within a small cage.

The behaviour of iodate in solution is more complex. In the aqueous phase the iodate ion has been considered as unhydrated. However, Nightingale *et al.*<sup>31</sup> provided evidence, by comparing the limiting equivalent conductances and viscosity coefficients for the iodate and periodate ions, to indicate the presence of an interaction between iodate ions and water molecules. Water molecules maintain a tetrahedral skeleton;<sup>32, 33</sup> the interionic distances of the crystal lattice structure of the solute are assumed to be fixed in solution. According to Fajans *et al.*,<sup>34</sup> most of the ions studied here fit into the water structure. The apparent volumes at infinite dilution for  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cl}^-$  and  $\text{OH}^-$  are 1.7, 8.4, 18.0 and 4.8  $\text{cm}^3$  per equiv., respectively, while that of the water molecule is 18.0  $\text{cm}^3$  per equiv. The ions having almost equal volumes can replace water molecules in the tetrahedral skeleton, and ionic forces will cause the interpenetrating water molecules surrounding the ion in the first and second spheres to approach each other and occupy a smaller volume than in pure water. However, the effect of ions on water molecules not immediately in the hydration sphere, *i.e.* 'free' water, is to shift the thermodynamic equilibrium between the tetrahedral and close-packed water structure towards the latter.<sup>35</sup> Obviously the ions with largest volumes (25.1  $\text{cm}^3$  per equiv for  $\text{IO}_3^-$  and 40.5  $\text{cm}^3$  per equiv. for  $\text{CH}_3\text{CO}_2^-$ ) will deform the water structure from a regular arrangement.

From the above discussion it can be concluded that the probability of the recoil species encountering an additive increases with increasing concentration of the latter. At lower concentrations of additives the total number of added ions with respect to each target ion is four. However, in the case of periodate this number is raised to *ca.* 114 for  $\text{KCl}$ , 146 for  $\text{NaCl}$  and 104 for the acetates, respectively. The hydration numbers are 13 ( $\text{Na}^+$ ), 7 ( $\text{K}^+$ ), 5 ( $\text{Cl}^-$ ) and 11 ( $\text{OAc}^-$ ). The volume occupied by the ion atmosphere decreases with the increasing concentration and the water molecules will occupy a smaller volume than in pure water. The main ionic radii with respect to the total concentration decreases from 7.6 Å to *ca.* 3.7 Å. Therefore, the number of collisions required for the recoil atom to come to thermal equilibrium will diminish because of the higher mass of the additives. When cations are involved in the recoil reaction, the affinity of the cations for electrons (for example) may affect the oxidation state of the recoil atoms. Salts of different cations have different crystal structures except for the case of isomorphs. This difference in crystal structure is possibly the origin of the cation effect.

Bernal and Fowler<sup>36</sup> have concluded that the higher the temperature or the concentration of the dissolved electrolytes, the more looser will be the structure. In the frozen alkaline aqueous state the introduction of  $\text{IO}_3^-$  and  $\text{NaOH}$  forms lattice defects in the ice crystals; this may be one of the causes of the oxidation process during the ( $n, \gamma$ ) reaction. Normally 12 water molecules constitute a rhombohedral structure<sup>37</sup> of pure ice and the single crystal is found to be the tetragonal with  $a = 6.27$  Å and  $c = 5.79$  Å. This gives a qualitative idea of on the extent of packing

in the recombination process. In addition, hypothetical metastable species must be considered to explain the variation in retention.

The reaction that  $^{128}\text{IO}^-$  undergoes to form iodate is the oxidation reaction. The oxidizing species is the OH radical formed by the radiolysis of water. The OH radicals formed are concentrated around the 10 Å region, while the H atoms diffuse away to *ca.* 150 Å. High internal conversion in  $^{128}\text{I}$  is known to occur<sup>38, 39</sup> and the emitted X-ray and highly positively charged iodine atoms are also expected to produce OH radicals. In presence of chloride, the increase in retention can also be explicable in the light of the formation of oxidizing radicals such as OH and  $\text{Cl}_2^-$ . Also, it is known that the  $\text{Cl}^-$  ion is a OH radical scavenger;<sup>40</sup> as the concentration of  $\text{Cl}^-$  increases the OH radicals are scavenged. The formation of  $\text{Cl}_2^-$  ions by irradiation of the solution cannot be accounted for by the theory of diffusion for spurs.<sup>41, 42</sup> In the light of above model the  $\text{Cl}^-$  ions present in large concentrations in the iodate/chloride target may react with  $\text{H}_2\text{O}^+$  forming  $\text{Cl}_2^-$  as



The chloride and acetate additives may also be oxidized by OH radicals to hypochlorites and peracetates, which are strong oxidizing agents. At a higher concentration of acetate the deviation in activity distribution may be due to the association of acetate ions, which on reaction with OH probably form other compounds (*e.g.* acetone) rather than the peracetate ion.<sup>43</sup> Also, as the additive concentration increases the distance between the two ions decreases.<sup>44</sup> With the increase of additives the dimensions of the 'hot' volume will tend towards a more compact form, thus increasing the probability of more high-energy atoms being trapped within this small volume

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