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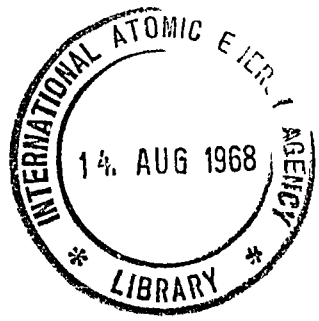

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ABSTRACT

Existing methods of separating fluorine-18 produced in reactor-irradiated lithium compounds are reviewed. Direct distillation of fluorine-18 from acidified lithium solutions and absorption with the minimum of condensation is shown to be the most satisfactory process for low activities. For remote operation the fluorine-18 is separated by absorption on a column of hydrous zirconium oxide followed by elution on passage of an alkaline solution to hydrolyse the complex fluoride. A lower recovery level for fluorine-18 on zirconium oxide is compensated by the speed at which this separation can be carried out. The irradiation of lithium hydroxide is recommended for fluorine-18 production. A procedure is described in detail for the separation of fluorine-18.

CONTENTS

1. INTRODUCTION

Fluorine has a number of radioactive nuclides of which fluorine-18 has the longest half-life (112 minutes). This half-life is relatively short in comparison with other nuclides used as radioactive tracers and effectively limits the period over which tracer experiments may be carried out with radioactive fluorine. Problems in the production of fluorine-18 arise from the necessity to separate and purify the radioisotope in the shortest possible time so that it may be available for tracer experiments at sites remote from the production scene.

2. REVIEW OF FLUORINE-18 PRODUCTION BY REACTOR IRRADIATION

2.1 Nuclear Reaction

The only useful method of obtaining fluorine-18 from reactor irradiation is the $^{6}Li\ (n,\alpha)t$; $^{16}O\ (t,n)\ ^{18}F$ sequence of reactions. To minimise energy dissipation of the tritons (maximum energy 2.73 MeV) by interaction with other nuclei, the lithium and oxygen are contained in the same crystal lattice. Severe self-shielding occurs when highly enriched lithium-6 compounds are used and the optimum thickness for a lithium-6 compound may be deduced from the information given by Stang (1965) to be about 1 mm. The level of fluorine impurity in enriched lithium compounds is generally lower than in commercially available lithium compounds so fluorine-18 can be obtained with a higher isotopic abundance from the enriched compound.

The amount of fluorine-18 produced by the reactor irradiation of compounds of lithium (at natural abundances) and oxygen has been observed to increase with the ratio of the number of oxygen atoms to the sum of the atomic numbers of the atoms in the compound (Bresesti et al. 1963). Although lithium nitrate is superior in this respect lithium carbonate is usually preferred because of its greater stability to reactor irradiation. Lithium hydroxide which has a value for this ratio equal to the carbonate must be considered as an alternative for fluorine-18 production.

A chemical separation from lithium salts and tritium is usually necessary. The method devised should also remove contaminating activities such as chlorine-38 and sodium-24 arising from neutron activation of impurities in the irradiated lithium compound.

2.2 Separation Methods for Fluorine-18 from Irradiated Lithium Compounds

Fluorine-18 was first separated by a co-precipitation method from a solution of an irradiated lithium salt followed by the distillation of the nuclide from a

sulphuric or perchloric acid solution of the precipitate (Knight et al. 1951, Bernstein and Katz 1953, Banks 1955, Münze and Baraniak 1960, Ericsson and Hammarstroem 1964, Nagy and Berei 1964). This technique will separate fluorine-18 from contaminating radioactive anions and has been recommended for the preparation of sodium fluoride (^{18}F) (Stranks 1963). However it is a comparatively awkward procedure to carry out remotely.

Fluorine-18 has been directly distilled from a solution of an irradiated lithium compound in sulphuric acid (Thomas et al. 1965). Only tritium and chlorine-38 were possible contaminants in the distillate.

Beg and Brown (1963) dissolved irradiated lithium oxide in water and passed the lithium hydroxide solution through a cation exchange column in the hydrogen form. Fluorine-18 was recovered in the eluate as hydrofluoric acid. However small amounts of fluoride will form complex fluoride cations (e.g. AlF^{2+}) with trace quantities of many elements. Retention of the complex cation by the cation exchange column will reduce the yield of separated fluorine-18.

Fluorine-18 was absorbed from slightly acid solutions of lithium salts on a column of chromatographic alumina (Stang et al. 1957, Bresesti et al. 1963, Shibata 1964). The fluorine-18 was recovered by elution with an alkaline solution. This is the separation process used at Brookhaven National Laboratory (Stang 1964) and the Japan Atomic Energy Research Institute.

The removal of fluoride ions from water by alumina has been used for a number of years (Boruff 1934, Fink and Lindsay 1936, Savinelli and Black 1958) but the absorption of fluorine-18 from strongly acid lithium solutions has been observed to be incomplete (Shibata 1964). Elution of an alumina column with sodium hydroxide produced fluorine-18 which contained significant quantities of aluminium (Shibata 1964). The absorption of fluorine-18 on to magnesium oxide has been attempted but recovery of the absorbed fluorine-18 proved difficult (Bresesti et al. 1963).

2.3 Possible Development of Fluorine-18 Separation Methods

The major impurity in fluorine-18 after distilling into alkaline solution is tritium. This could be greatly reduced by a repeated evaporation to dryness (but not removed completely, as NaOT is formed). Some experimental modifications to the distillation procedure could expedite the overall separation.

An absorption and desorption behaviour of fluorine-18 similar to that with alumina could be expected with the hydrous oxides of zirconium, tin (IV) and

titanium. In acid solution the fluoride ion forms a complex with the the metal atom of the oxide and elution with alkali brings about the hydrolysis of the fluoride complex, resulting in the desorption of fluoride ions.

Zirconium forms the most stable complexes with fluoride ions (Paul 1955). Hydrous zirconium oxide which is commercially available (Bio Rad Laboratories) has been observed to have 'an enormous capacity for fluoride' (Kraus et al. 1956). Titanium also forms very stable complex fluorides (Kleiner 1952) but in acid solution the surface potential of the hydrous oxide is negative (Johansen and Buchanan 1958), so the anion exchange properties will be very weak. Hydrous stannic oxide has been found to have a positive surface potential lower than for alumina and also ion exchange properties similar to alumina (O'Connor and Buchanan 1953). The stability of complex stannic fluorides has been reported to be comparable to that of aluminium fluorides (Babko 1959).

2.4 Chemical Form of Separated Fluorine-18

Many complex fluorides exist in acid solution but most are hydrolysed as the pH is increased. Low concentrations of fluosilicate (Ryss 1951) and fluoborate (Ryss and Donskayen 1959) are hydrolysed in alkaline solution. The species AlF^{2+} is easily hydrolysed with an equilibrium constant of 10^{11} for the reaction:

$$AlF^{2+} + 3(OH)^- = Al(OH)_3 + F^-,$$

but the hydrolysis of complex aluminium fluorides is suppressed for ratios of fluoride ions: aluminium ions which exceed 3:1 (Kleiner 1950).

Fluorine-18 distilled from glass and absorbed in sodium hydroxide solution will be in the form of fluoride ions. Elution with alkali from a column of a hydrous metal oxide proceeds by hydrolysis of a complex fluoride and fluorine-18 will be found as fluoride ions in the eluate.

3. EXPERIMENTAL

3.1 Lithium Compounds Irradiated

Analytical grade lithium carbonate, hydroxide and sulphate, and lithium carbonate with a lithium-6 abundance of 95 per cent, were irradiated. These materials contained only small amounts of chloride and aluminium ions and a wide variation in fluorine content (Table 2).

3.2 Irradiations

Up to one-gram quantities of the lithium compounds sealed in quartz ampoules were irradiated in a 6 HGR facility in HIFAR for periods of up to 6 hours at a

nominal thermal neutron flux of 9 x 10^{12} n/cm^2/sec. The amount of fluorine-18 produced in each sample (Table 1) was estimated from the decay curves obtained from measurements in a calibrated 4π gamma ionisation chamber.

In order to determine the flux depression caused by the enriched lithium target the ^{24}Na activity obtained by irradiating 20 mg of sodium carbonate was compared with the ^{24}Na activity obtained by irradiating 20 mg of sodium carbonate mixed with 100 g of lithium carbonate (95 per cent ^{6}Li) under identical conditions. The reduced ^{24}Na activity arising in the sodium carbonate mixed with lithium carbonate indicated a flux depression of 50 per cent.

3.3 Separation of Fluorine-18 by Distillation

The glass distillation apparatus consisted of a 50 ml round-bottom flask heated by an infra-red heater, and a tube to bubble air through the contents of the flask. The air sparge from the flask carrying the distilled fluorine-18 passed directly through a small still head into 1 ml of 0.1 M sodium hydroxide contained in a receiver designed for efficient contact of the air current with the alkaline solution. To retain the fluorine-18 in the minimum volume no condenser was used.

The ampoules of the irradiated lithium compound were broken and the fragments and contents transferred to the distillation flask. Quantities of lithium compounds weighing up to 4 g were used. Twenty ml of approximately 50 per cent sulphuric acid was added and distillation started.

With 1 g of lithium hydroxide approximately 75 per cent of the fluorine-18 was distilled into the receiver in 30 minutes. Distillation was more rapid from higher concentrations of lithium salts; for example, almost 80 per cent of the fluorine-18 was in the receiver after 20 minutes distillation when 4 g of lithium hydroxide were used. After the distillation the receiver solution was neutralised to pH 7-8 with dilute hydrochloric acid. Much of the initial tritium in the still was found in the receiver, so the fluorine-18 solution was evaporated to dryness twice to reduce the tritium level. This resulted in a total tritium content of 0.2 µCi in the separated fluorine-18 when the initial tritium level in the still had been 10 µCi per gram of lithium carbonate.

Normally no lithium could be detected in the distillate by flame photometry. As a small sample of the fluorine-18 solution was diluted for the determination the detection limit was 50 p.p.m. Occasionally some lithium was detected when irradiated lithium carbonate had dissolved with much effervescence.

3.4 Separation of Fluorine-18 from Lithium by Ion Exchange

An aqueous solution of 1 g of irradiated lithium hydroxide was passed through a column of the cation exchange resin Bio Rad AG50WX8 (hydrogen form) to remove all lithium ions. It was expected that fluoride ions would not be absorbed on the column but more than 90 per cent of the fluorine-18 was retained on the column and failed to be recovered in the eluate.

To demonstrate the influence of fluoride complexing ions on fluorine-18 recovery, quantities of aluminium nitrate were added to 0.15 M lithium chloride solutions at pH 1-2 containing some fluorine-18. Each solution was passed through a column of the cation exchanger Bio Rad AG50WX8 (hydrogen form) to remove all lithium from solution. The proportion of fluorine-18 retained by the column partly depended on the fluorine:aluminium ratio (Table 2).

3.5 Separation of Fluorine-18 on Hydrous Metal Oxide

3.5.1 Materials

Hydrated zirconium oxide, 50-100 mesh, was supplied by Bio Rad Laboratories, and neutral chromatographic alumina was supplied by Woelm. Hydrous stannic oxide was prepared (Merz 1959) by the reaction of nitric acid on granulated tin. The washed tin oxide was finally dried in a vacuum oven at 100-110°C for two hours, then sieved to 60-85 mesh. Hydrous lead dioxide was prepared by anodic deposition onto platinum from lead nitrate solution at 60°C, then dried at 100-110°C in a vacuum oven for 1 hour. Hydrous titanium dioxide was prepared by precipitation from homogeneous solution with urea. The washed material was dried at 100-110°C in a vacuum oven for two hours and then sieved to remove fines.

3.5.2 Partition experiments

The comparative uptake of fluorine-18 on hydrous metal oxides at ambient temperatures was examined by mixing an approximately 100 mg quantity of each oxide with 20 ml hydrochloric acid solution containing fluorine-18 with a fluoride ion content of less than 1 p.p.m. and shaking until equilibrium was reached. The amount of fluorine-18 absorbed by the oxide was estimated from the reduction in the activity of the solution. As only small quantities of each oxide were used the distribution coefficients obtained (Table 3) must be only considered as a guide to the relative absorption of fluoride by these oxides.

3.5.3 Column experiments

These experiments were carried out with glass columns 10 mm in internal diameter containing the hydrous oxide supported on a sintered glass plate. A

plug of quartz wool was packed on top of the oxide layer to prevent disturbance of the bed. Each hydrous oxide was washed in turn with 1-2 M ammonia solution, water and 1 M hydrochloric acid before being transferred to the column as a slurry in water.

The separation of fluorine-18 from acid solution onto columns containing 1 or 2 grams of alumina was examined. The retention of the fluorine-18 by the alumina was variable and appeared to depend on the lithium chloride and hydrochloric acid concentrations and the volume of the solution (Table 4). More than 75 per cent of the tritium in the column feed solution passed through the column without being absorbed. More than 90 per cent of the fluorine-18 was absorbed by a 1 g column of hydrous zirconium oxide, from a solution of 4 M lithium chloride and 0.3 M hydrochloric acid. The zirconium oxide column suffered little attack during the fluorine-18 absorption as only 15 p.p.m. of zirconium was found in the raffinate. Less than 0.1 p.p.m. of zirconium was found in the solution of fluorine-18 removed from the column by 1-2 M ammonia solution. Up to 50 ml of 0.15 M sodium hydroxide solution was required to elute the fluorine-18 absorbed on a 1 g column of zirconium oxide.

The absorption of fluorine-18 by hydrous stannic oxide is very efficient from acid solutions containing high lithium concentrations but strong ammonia solutions are necessary to remove the absorbed fluorine-18. Only 10-15 per cent of the initial tritium was recovered with the fluorine-18 from columns of tin and zirconium oxide. This tritium was reduced to an insignificant level by evaporation to dryness.

Small amounts of lithium ions were retained by aluminium, zirconium and tin oxide columns during absorption of the fluorine-18. Lithium was easily removed by passing 10 ml of 0.1 M hydrochloric acid through the column although this treatment tended to remove fluorine-18 from the aluminium oxide column.

The relative performance of 1 g quantities of aluminium, tin and zirconium oxides (Table 5) favours the use of zirconium oxide in obtaining a fluorine-18 solution relatively free from complexing metal ions and higher flow rates were found for this material. Four batches of fluorine-18 for animal experiments were prepared by separation on a zirconium oxide column which was reused for each separation. Only a small decrease in recovery of fluorine-18 was observed with the repeated use of the column.

3.6 Analytical Methods

Measurements of fluorine-18 activity were made in a calibrated 4π gamma ionisation chamber and the radioisotopic purity of the nuclide was examined with a multichannel gamma spectrometer. Tritium was estimated by liquid scintillation counting after decay of fluorine-18. For distribution measurements fluorine-18 samples were counted in a well crystal gamma counter.

The fluoride ion concentration in a solution of fluorine-18 separated by distillation or on hydrous zirconium oxide was estimated by the method of Valach (1961) without a further distillation separation. This procedure involves the addition of a zirconium solution to a pure fluoride sample and the spectrophotometric measurement of the uncomplexed zirconium. The zirconium content of the samples from the zirconium oxide columns was a negligible proportion of the zirconium added in the analysis.

Some modification was necessary to reduce the time required for analysis. Valach recommended heating the sample with the zirconium solution at 60°C for 20 minutes and this heating period was found to be necessary. However, the subsequent procedure of cooling for 40 minutes then waiting one hour before measurement of the uncomplexed zirconium was found to be not essential and the samples were cooled rapidly in cold water and the zirconium measured immediately.

4. DISCUSSION

Since fluorine-18 has a relatively short half-life (112 minutes) the commercial success, or otherwise, of a separation procedure must be judged in terms of the time taken for processing. In fact almost equal periods are required for the separation of fluorine-18 by distillation or by using a hydrous zirconium oxide column, and both methods result in a solution containing some tritium.

The separation of fluorine-18 by distillation in glass from sulphuric acid solutions of irradiated lithium compounds is probably the better separation method although considerable care is necessary to avoid bumping if the separation is being carried out remotely. Yields are high, no fluoride complexing elements other than those in glass are introduced and the separated fluorine-18 is obtained in a small volume which can be rapidly evaporated to reduce the tritium content.

Fluorine-18 is absorbed on a column of a hydrous metal oxide as a complex fluoride of the metal atom of the oxide; the ease of elution of the fluorine-18 depends on the ease of hydrolysis of the complex fluoride. Less alkali is

required to elute fluorine-18 from a column of aluminium oxide than from stannic or zirconium oxides where the complex fluorides are more difficult to hydrolyse. Elution with alkali strips anions from hydrous metal oxide columns by an ion exchange mechanism. As the isoelectric point of the stannic oxide is low (pH approximately 4.8, Kraus et al. 1956), absorbed anions may be removed quite rapidly by washing with water before fluorine-18 is eluted. Thus fluorine-18 may be separated relatively free of ions such as chloride (Table 5). Apart from this application no advantage is apparent in the use of hydrous stannic oxide.

The overall performance of a column of 1 g of hydrous zirconium oxide is superior to aluminium oxide columns. The yield of fluorine-18 separated on a zirconium oxide column is lower than when an alumina column is used. However this is offset by the fact that a smaller column is required for efficient absorption of the fluorine-18 and greatly increased flow rates are possible. A zirconium oxide column has the advantage of introducing only very small traces of zirconium and no other fluoride complexing agents into the separated fluorine-18 solution. This column can be used to absorb fluorine-18 efficiently from strongly acid solutions without impairing its function, so it is unnecessary to regulate closely the acidity of the solution from which fluorine-18 is absorbed.

When fluorine-18 is required for any particular experiment it should be taken into account that the chemical form depends on the nature and quantity of other ions present and on the pH of the solution. Also the specific activity of the fluorine-18 is determined mainly by fluorine content of the lithium compound irradiated, with some additional fluorine being introduced in the reagents. It can be seen (Table 2) that there was a wide variation in the fluorine levels in the lithium compounds irradiated in this work so it is advisable to determine the fluorine content of any lithium compound to be irradiated.

Lithium hydroxide containing lithium at natural abundances proved to be a most useful compound for the preparation of fluorine-18. More fluorine-18 per gram of compound was obtained for the hydroxide than for the carbonate (Table 2) which is not entirely in accordance with the relationship between fluorine-18 production and $\frac{\text{number of oxygen atoms}}{\Sigma \text{ atomic numbers}}$ proposed by Bresesti et al. (1963).

5. CONCLUSIONS

(1) When small quantities of fluorine-18 are required and remote techniques are unnecessary fluorine-18 is most efficiently separated from irradiated lithium compounds by distillation from sulphuric acid solutions.

(2) Millicurie quantities of fluorine-18 require remote or semi-remote handling during the separation process. The most suitable process is to absorb the fluorine from acid solution on a column of hydrous zirconium oxide. The fluorine-18 product is obtained by eluting with alkaline solution. A detailed production procedure is recommended in the Appendix.

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APPENDIX

RECOMMENDED PROCEDURE FOR THE SEPARATION OF FLUORINE-18 FROM IRRADIATED LITHIUM COMPOUNDS

SPECIAL APPARATUS

Glass column 10 mm diameter containing 1.00 g (accurately weighed) of hydrous zirconium oxide (Bio Rad HZO-1, 50-100 mesh) supported on a quartz wool pad and with a small plug of quartz wool on top of the oxide bed to reduce disturbance.

REAGENTS

1. Concentrated hydrochloric acid.
2. Hydrochloric acid approximately 10% $^{v}/_{v}$ dilution from concentrated acid.
3. Ammonia solution approximately 10% $^{v}/_{v}$ dilution from concentrated ammonia solution.
4. Standard 1 N sodium hydroxide solution.

PROCEDURE

1. Add water to the irradiated lithium compound at about 10 ml per gram of compound. Add sufficient concentrated hydrochloric acid to neutralise any basic compound and to give a final acid concentration of between 0.1 and 1.0 N.
2. Pass the acid solution through the column allowing it to flow freely. Discard eluate. ^{18}F is retained on the column.
3. Wash the column twice with 10 ml of 10% hydrochloric acid to remove any lithium ions.
4. Wash the column twice with 15 ml of water.
5. Elute the fluorine-18 from the column with three 10 ml portions of 10% ammonia solution.
6. Add accurately 2.3 ml of 1N sodium hydroxide solution to the eluate and evaporate to dryness to remove excess ammonia and most of the tritium.
7. Add 2-3 ml of water to the residue and repeat the evaporation.
8. Dissolve the residue in 5-10 ml of water. Adjust the pH to 8-10 with dilute hydrochloric acid and dilute to 15 ml with water. This product now contains fluorine-18 as fluoride ions in isotonic saline.

TABLE 1

PROPERTIES OF THE LITHIUM COMPOUNDS IRRADIATED

Properties	Lithium Carbonate	Lithium Carbonate 95% ^{6}Li	Lithium Hydroxide	Lithium Sulphate
Fluorine content (p.p.m.)	500	< 4	35	9
Chloride Ion (p.p.m.)	< 20	< 10	< 10	25
Aluminium (p.p.m.)	< 10	-	< 5	20
Activity (mCi) of $^{18}F/g$ of lithium compound for 6 hour irradiation at approx. 9 x 10^{12} $n/cm^2/sec.$	4.5	41*	5.3	3.3
Activity (mCi) of $^{18}F/g$ ^{6}Li	330	230	250	350

* Flux depression in sample

TABLE 2

RELATION OF FLUORINE: ALUMINIUM RATIO TO RETENTION OF FLUORINE-18 ON CATION EXCHANGER DOWEX 50

Ratio F : Al	Percentage of ^{18}F Retained on Resin
6	73
1.5	94
0.6	93

TABLE 3

DISTRIBUTION COEFFICIENTS FOR FLUORINE-18 BETWEEN SOLUTION AND HYDROUS METAL OXIDES AT AMBIENT TEMPERATURE

$$\text{Distribution Coefficient} = \frac{^{18}F \text{ absorbed per gram of hydrous oxide}}{^{18}F \text{ per ml of solution at equilibrium}}$$

Solution	PbO_2	Al_2O_3	ZrO_2	TiO_2	SnO_2
0.1 M HCl	90	70	300	N.D.	300
0.01 M HCl	40	40	100	N.D.	300
HCl pH 4.2	N.D.	200	100	50	400
1.5 M Ammonia	N.D.	5	8	10	2

TABLE 4

ABSORPTION OF FLUORINE-18 ON TO ALUMINA COLUMNS 10 mm IN DIAMETER

Solution		^{18}F Absorbed (%)	
(M) LiCl	(M) HCl	1 g alumina	2 g alumina
1.1	0.1	57	65
1.1	0.2	55	53
1.1	0.5	53	N.D.
1.1	1	54	N.D.
1.1	2	30	53
0.05	0.1	90	N.D.
0	1	96	N.D.

TABLE 5

COMPARISON OF FLUORINE-18 SEPARATION ON 1 g COLUMNS OF THE HYDROUS OXIDES OF ALUMINIUM, ZIRCONIUM AND TIN (IV)

Solution Passed Through Column	Distribution of ^{18}F on 1 g Column of the Hydrous Oxide (%)					
	Aluminium		Zirconium		Tin	
	On Column	In Eluate	On Column	In Eluate	On Column	In Eluate
10 ml 0.05 M LiCl and 0.1 M HCl	96.8	3.2	96.8	3.2	96.9	3.1
10 ml 0.1 M HCl	86.3	10.5	95.1	1.7	95.5	1.4
2 x 10 ml water	81.4	4.9	95.1	0.1	95.5	0.1
10 ml 1-2 M ammonia	7.3	74.1	31.9	63.2	35.2	60.3
10 ml 1-2 M ammonia	4.0	3.3	21.5	10.4	28.9	6.3
Concentration of metal ion in ^{18}F ammonia solution	50 p.p.m.		0.1 p.p.m.		3 p.p.m.	
Concentration of chloride ion in ^{18}F ammonia solution	N.D.		3 mg/ml		0.15 mg/ml	