THE DETERMINATION OF 210 POLONIUM IN URINE AND OTHER BIOLOGICAL MATERIALS

CHEMICAL SERVICES DEPARTMENT

OPERATIONS BRANCH

WINDSCALE

SUMMARY Organic matter is destroyed by wet oxidation, and polonium is co-precipitated with tellurium carrier by reduction with sodium hypophosphite. The precipitate is dissolved and tellurium is removed by precipitation using hydrazine hydrochloride. After electrochemical deposition from solution on to silver foil the activity due to 210 polonium is determined in an alpha scintillation counter.

APPLICABILITY:

General	Accuracy:	Bias – 30%	) Based on 22 determinations by 20
	Precision:	3 σ ± 65%	) operators using 500 ml. portions of urine to which 11 d.p.m.* portions of 210 polonium had been added.
	Accuracy:	Bias – 34%	) Based on 10 determinations by one
	Precision:	3 σ ± 102%	) operator using 500 ml. portions of urine to which 5 d.p.m. portions of 210 polonium had been added.

Concentration Range: Up to 6 x 10^4 d.p.m. 210 polonium on disc.
Limit of Detection: 5 d.p.m. 210 polonium.

Specific To the determination of 210 polonium in urine, blood, faeces and other biological samples.

HISTORY:

Based on A.E.R.E. HP/R.627, a private communication from D. A. Lambie of R.C.C. Amersham and on development work at C.I., Windscale by Celia J. Banks, A.E. Dyke and F.B. Sumner.

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* d.p.m. = disintegrations per minute

UNITED KINGDOM ATOMIC ENERGY AUTHORITY

INDUSTRIAL GROUP

RISLEY WARRINGTON LANCASHIRE

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DISCLAIMER

A. OUTLINE OF METHOD

The sample is digested, first with nitric and sulphuric acids and then with perchloric acid to remove all traces of organic matter. The residue is dissolved in water and potassium tellurate is added as a carrier. Polonium is co-precipitated with metallic tellurium on addition of sodium hypophosphite and this precipitate is dissolved in a saturated solution of bromine in hydrochloric acid; tellurium is precipitated by the addition of hydrazine hydrochloride and is removed by filtration. The acidity and volume of the filtrate are adjusted and polonium is deposited electrochemically on silver foil. The activity of the deposited polonium is determined in an alpha scintillation counter and is counted against a standard plutonium source; differences in energies, backscatter and geometry are accounted for in the overall bias of the method.

B. SPECIAL REAGENTS REQUIRED

1. **Bromine/Hydrochloric Acid**

Saturated solution of analytical reagent quality bromine in 11M analytical reagent quality hydrochloric acid.

2. **Potassium Tellurate Solution 1 ml. = 1 mg Te**

Dissolve 0·5 g of powdered tellurium in 40 ml. of the saturated bromine/hydrochloric acid reagent. Evaporate to about 10 ml. and dilute to 100 ml. with distilled water. Add 200 ml. of 2% w/v potassium hydroxide solution; allow the white precipitate which forms to re-dissolve and dilute to 500 ml. with water.

3.	**Fuming Nitric Acid**	Pure
4.	**Nitric Acid 16M**	
5.	**Sulphuric Acid 18M**	
6.	**Hydrochloric Acid 11M**	Analytical Reagent Quality
7.	**Hydrochloric Acid 6M**	
8.	**Hydrochloric Acid 0·5M**	
9.	**Sodium Hydroxide Solution 30% w/v**	
10.	**Perchloric Acid 60%**	
11.	**Sodium Hypophosphite**	
12.	**Hydrazine Hydrochloride**	
13.	**Perspex Cement**	

Dissolve perspex shavings in chloroform.

C. SPECIAL APPARATUS REQUIRED

1. Polished Silver Discs – 2$\frac{3}{8}$ in. diameter, 0·004 in. gauge.
2. Mechanical Stirrer with Glass Paddle
3. Alpha Scintillation Counter

D. EXPERIMENTAL

Procedure

1.1 Urine Samples

Measure the volume of the sample [Note (a)] and transfer the whole sample including solid matter to a suitable beaker. Take a sample portion for the determination of creatinine [Note (b)]. Rinse the sample bottle with two 10 ml. portions of 16M nitric acid, adding the washings to the sample. Add 25 ml. of 18M sulphuric acid to the sample. Transfer the beaker to the fume cupboard and add carefully, with stirring, about one fifth of its volume of fuming nitric acid.

1.2 Blood Samples

Measure the volume of the sample and transfer to a 1 litre beaker; cautiously add a mixture of 100 ml. of fuming nitric acid and 25 ml. of 18M sulphuric acid, using portions of the acid mixture to rinse the sample container and the measuring vessel. Allow the acidified sample to digest in the cold with occasional stirring until frothing has ceased.

1.3 Faeces Samples

Transfer the weighed sample to a 1 litre beaker and cautiously add a mixture of 100 ml. of fuming nitric acid and 25 ml. of 18M sulphuric acid, using a portion of the mixture to rinse the sample container; allow the acidified sample to digest in the cold with occasional stirring until frothing has ceased.

1.4 Other biological samples

Transfer the sample to a suitable beaker and add a mixture of 100 ml. of fuming nitric acid and 25 ml. of 18M sulphuric acid, using a portion of the mixture to rinse the sample container.

Notes

(a) The sample should be collected into 25 ml. of 16M nitric acid to avoid adsorption losses of polonium on the glass.

(b) See IGO-AM/W-69.

Procedure

2. By evaporation, reduce the contents of the beaker to a volume of about 100 ml. [Notes (c) and (d)]

3. Transfer the solution to a 4½ in. silica dish, washing the beaker with two 10 ml. portions of 16M nitric acid and adding the washings to the dish. Heat on a sand-bath at a temperature not exceeding 250°C until nitrous fumes cease to be evolved and the sulphuric acid darkens, due to charring. Remove the dish from the sand bath and allow to cool.

4. Cautiously add 10 ml. of fuming nitric acid and heat the dish again on the sand bath until nitrous fumes cease to be evolved. Remove the dish from the sand bath and allow to cool.

5. Cautiously add 10 ml. of 60% perchloric acid [Note (e)]. Heat the dish on the sand-bath until perchloric acid fumes cease to be evolved. If the residual sulphuric acid still shows evidence of charring, continue treatment with small portions of fuming nitric acid until all organic matter has been destroyed and the colour of the residual sulphuric acid is pale straw. Allow the solution to cool.

6. Transfer the solution to a 600 ml. beaker containing about 200 ml. of water; rinse the dish with water and add the rinsings to the beaker [Note (f)].

7. Add 10 ml. of potassium tellurate solution to the beaker and heat the contents almost to boiling. Cautiously add solid sodium hypophosphite until a black precipitate of tellurium begins to form; add 1–2 g of the reagent in excess. [Note (g)]

8. Cover the beaker with a cover glass and boil gently for a short time to coagulate the precipitate. [Note (h)].

9. Rinse the cover glass with water, adding the rinsings to the beaker. Filter whilst hot through a 12·5 cm Whatman No. 40 paper, washing the precipitate with water. Retain the filtrate.

Notes

(c) Evaporation should be carried out in a fume cupboard.

(d) Excessive frothing should be avoided.

(e) Details of precautions necessary when handling perchloric acid will be found in textbooks of analytical chemistry or in B.S.1121A (1949)

(f) The final volume should be about 300 ml.

(g) If nitric acid is present in the solution, effervescence will take place on addition of sodium hypophosphite and a greater amount of the reagent than normal will be required.

(h) Vigorous boiling may cause mechanical losses.

Procedure	Notes
10. Dissolve the precipitate through the paper by the dropwise addition of 5 ml. of the bromine/hydrochloric acid reagent [Note (i)] . Wash the paper with 5 ml. of 6M hydrochloric acid [Note (i)] . Warm the solution to remove the bulk of the excess bromine.	(i) Add the reagent around the edges of the paper, allowing the paper to drain after each addition of about 30 drops.
11. On the filtrate obtained in para. 9, carry out a further tellurium co-precipitation by the procedures described in paras. 7–10.	
12. Combine the two solutions obtained as in para. 10 and dilute to about 40 ml. with water; add 2 g of hydrazine hydrochloride, cover the beaker with a cover glass and boil gently for a few minutes to coagulate the tellurium precipitate. Filter through a 12•5 cm Whatman No. 40 paper, washing the precipitate with 0•5M hydrochloric acid and collecting the filtrate in a 250 ml. squat beaker.	
13. Heat the filtrate for at least ten minutes; should a further precipitate of tellurium appear, filter the solution once more; allow the filtrate to cool.	
14. Add 3 drops of phenolphthalein indicator solution and neutralise the filtrate with 30% sodium hydroxide solution. Immediately add 4•5 ml. of 11M hydrochloric acid and dilute to about 100 ml. with water.	
15. Prepare a silver disc by coating the whole of one surface and part of the other with perspex cement so that only a clear central circle of 1•5 in. diameter remains. Determine the alpha activity of the disc using an alpha scintillation counter. [Note (j)] .	(j) The background activity is normally counted for 2 hours and should not exceed 15 c.p.h. *
16. Place the silver dish in the solution as prepared in para. 14 with the uncoated surface facing upwards. Heat the beaker on a hot plate so regulated that the solution temperature is maintained at 80°C. Carry out continuous mechanical stirring at 80°C for 1½ hours. [Notes (k) and (1)] .	(k) Any blackening of the disc shows that tellurium has not been completely removed; co-deposition of tellurium with polonium will cause absorption losses during the radiometric count. (1) Deposition of polonium on silver is extremely variable and the plating conditions must be rigidly followed.

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* C.p.h. = Counts per hour

Procedure

17. Remove the disc from the solution, wash with water and allow the disc to dry. Count in an alpha scintillation counter, correcting for background (para. 15) and counter efficiency.

18. Calculate the activity on the disc [Notes (m) and (n)])

$$\text{d.p.m. } {}^{210}\text{Po} = \frac{D\,(N - B_1)}{60\,(S - B)}$$

where D = disintegrations per hour of the standard plutonium source.

N = counting rate (c.p.h.) of the sample.

S = counting rate (c.p.h.) of the standard plutonium source.

B = background of counter using clean tray.

B_1 = background using tray used for sample.

19. Carry out a control determination by the procedures outlined above on a similar biological sample obtained from a subject known to have no contact with 210 polonium. Apply the result of this determination as a blank correction.

20. Calculate the 210 polonium content of the sample, corrected for bias, and express the result as d.p.m./g or ml. as appropriate. [Note (o)]. The activity in $\mu\mu$c can be calculated by dividing d.p.m. by 2·22.

Notes

(m) The alpha activity is counted against a standard plutonium source. Differences in energies, back scatter and geometry are accounted for in the overall bias of the method.

(n) A constant check on the accuracy, precision and reliability of the method should be maintained by analysing standard samples of 210 polonium in urine at frequent intervals. The characteristics of the method are assumed to be the same for all types of biological samples.

(o) Urine samples may also be expressed as d.p.m./2g of creatinine by the expression

$$\text{Activity } ({}^{210}\text{Po}) = \frac{R.\ 2000}{C}\ \text{dpm}$$

where C = creatinine content of sample in mg.

R = activity on disc. (para. 18).