Short Communications

On the Half-Life of [33]P

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[33]P was discovered by three teams independently, *viz.* Sheline *et al.*[1], Jensen *et al.*[2] and Westermark[3], but unfortunately there exists a discrepancy in the half-life determinations on this isotope. In his first experiments[3], Westermark found a half-life of 25 ± 2 days by studying the beta activity from a mixed sample of ^{32}P and ^{33}P and correcting for the amount of ^{32}P by using absorbers of aluminium. Later measurements[4], begun with a sample containing about 70 % [33]P and 30 % [32]P, gave 25.4 ± 0.3 days. Sheline *et al.*[1] give the value 25 ± 2 days, while Jensen and Nichols[2] in their first publication on the subject give 24.8 ± 0.5 days. Further experiments[5], where the amount of ^{33}P was determined from its area in a beta-spectrum from a mixture of ^{32}P and ^{33}P, gave a half-life of 24.4 ± 0.2 days.

Russell[6] found a half-life for ^{33}P of 25 ± 1 days by calculating it from an analysis of the decay curve of a [32]P—[33]P mixture taken with a Lauritsen electroscope.

As can be seen there is a discrepancy between the two most accurate figures, namely those given by Westermark[4] and by Nichols and Jensen[5]. As ^{33}P will probably be important in the future[7], especially for biochemists, it is of interest to determine the half-life of ^{33}P with a greater accuracy.

In this laboratory it was found that ^{33}P was formed almost free from ^{32}P upon irradiating sulphur far out at the end of the thermal column in the Norwegian D_2O-reactor[8]. It was also found that large amounts of sulphur (1—10 kg) could be irradiated between the concrete wall and the reflector and that a P activity containing about 25 % [33]P was thereby formed.

Preparation of the ^{33}P samples. In order to produce a sample of ^{33}P with a low ^{32}P-content, two batches of sulphur were irradiated. A 200 g sample of sulphur was irradiated in the outmost part of the JENER reactor for one month and then left to age for about two months. Another sample consisting of 1 kg was irradiated between the reflector and the concrete wall for one month and then left to age for about six months.

The radioactive phosphorus was separated from the sulphur by dissolving in CS_2 and filtering through a filter paper. About 50 % of the P activity was then found in the amorphous sulphur lying on the filter paper as shown by earlier experiments in this laboratory[3,9]. The PO_4^{3-} was extracted from the amorphous sulphur by boiling with 5 N HNO_3 and PO_4^{3-} carrier. The phosphate was then precipitated from the filtered solution as a coprecipitate with lanthanum hydroxide formed by adding lanthanum nitrate and then ammonia. The lanthanum hydroxide (after filtering and washing) was dissolved in HCl and the solution purified, after evaporation and dissolution in 0.02 N HCl, by cation exchange and then by several evaporations with H_2O_2 and a final evaporation with H_2SO_4. The residue was then taken up in a small volume of water and transferred to a lucite cup which had been painted with graphite in ethanol (a commercial mixture of "aqua-dag") and evaporated to dryness. The cup was covered with pliofilm (thickness 0.36 mg/cm^2).

Activity measurements. Beta activity measurements were performed over a period of about half a year. During all measurements, which were made with an end window GM-tube (1.4 mg/cm^2), the cup containing the sample was connected electrically to ground. The distance between the cup and the window of the tube was about 3 cm. Measurements with absorbers consisting of 10.1 mg/cm^2 Al and 55.2 mg/cm^2 Al were also made. Each measurement involved more than 15 000 counts
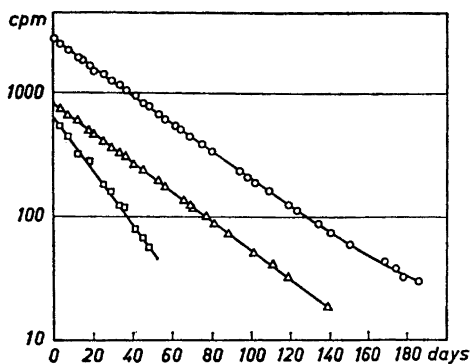
Fig. 1. Activity measurements of ^{33}P sample 2: O No aluminium absorber; △ 10.10 mg/cm^2 aluminium absorber; □ 55.22 mg/cm^2 aluminium absorber (scale raised by factor of 10).
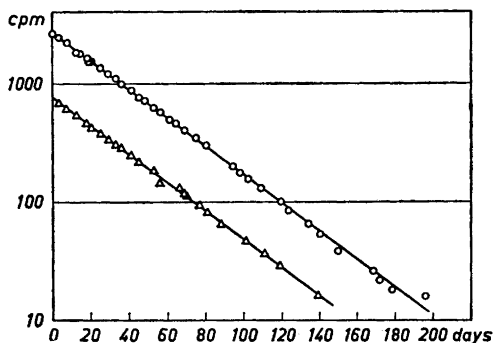

Fig. 2. Decay curves for ^{33}P sample 2: O No aluminium absorber; △ 10.10 mg/cm^2 aluminium absorber.

and usually more than 40 000. A non-aged ^{32}P source was measured both without any absorber and also with 10.1 mg/cm^2 Al and 55.2 mg/cm^2 Al absorbers in the same geometry as that for the ^{33}P-samples. The source was prepared from a carrier-free ^{32}P sample obtained from AERE, Harwell. This sample had been separated from sulphur shortly after the irradiation and probably contained some ^{33}P although not much. As a reference, a standard preparation of ^{60}Co was used.

Results. Fig. 1 shows the results obtained for sample 2 prepared from the 0.2 kg batch. It contained the largest fraction of ^{33}P (the rest being ^{32}P). The figures have been corrected for dead time losses and background. By means of the ^{60}Co standard which has a maximum beta energy (310 keV) not very far from that of ^{33}P (250 keV), corrections for changes in the apparatus, for instance due to changes in the GM-tube or the high voltage, and for changes in temperature and air pressure have been made. The activity measured with the absorber consisting of 55.2 mg/cm^2 Al should originate from ^{32}P alone since there is very little possibility of activities other than ^{32}P, ^{33}P and ^{35}S being present in the sample and since the ^{33}P and ^{35}S beta radiations have a shorter range than 55.2 mg/cm^2 apart from a small amount of bremsstrahlung. The measurements on the almost pure ^{32}P-sample (less than 1 % ^{33}P) with different absorbers gave a count-

ing rate ratio $I_0/I_{55.2}$ (index refers to aluminium absorber used, index 0 means no absorber) of 1.218 and $I_{10.1}/I_{55.2}$ of 1.176. From the 55.2 mg/cm^2 curve for the sample and the above data, the GM response from the ^{32}P activity could be calculated and corrected for.

As can be seen in Fig. 1, the curve with no aluminium absorber for sample 2 bends off towards a longer half-life after about 150 days. It is very probable that this is
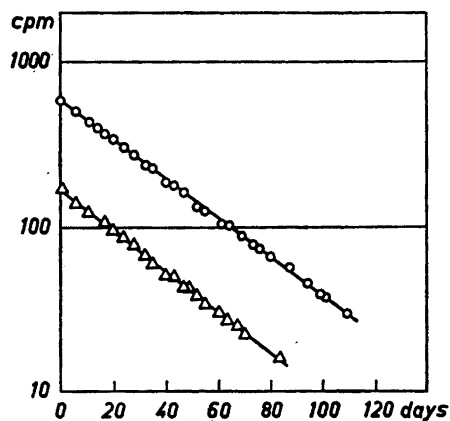

Fig. 3. Decay curves for ^{33}P sample 1: Q No aluminium absorber; △ 10.10 mg/cm^2 aluminium absorber.

caused by some ^{35}S being present in the sample despite the elaborate chemical procedures. The amount of ^{35}S giving rise to this longer half-life was calculated from the activity figures for the sample under the assumption that the half-life of ^{33}P is 25 days. It should be pointed out that this iterative procedure does not influence the main half-life determination because the correction for ^{35}S is rather small. From absorption curves for pure ^{35}S, corrections for ^{35}S could also be made for the 10.1 mg/cm^2 curve. The composition of the activity from the sample counted by the GM-tube with no aluminium absorber when the measurement started could thus be calculated to be: 95 % ^{33}P, 2.7 % ^{32}P and 2.2 % ^{35}S. The relative contributions of the radiation passing through the 10.1 mg/cm^2 absorber were 90 % ^{33}P, 9 % ^{32}P and 0.8 % ^{35}S.

Fig. 2 shows the ^{33}P decay curves, with no absorber and with 10.1 mg/cm^2 Al, obtained from Fig. 1 when corrections determined by means of the ^{60}Co standard and corrections for ^{32}P and ^{35}S had been made. The 10.1 mg/cm^2 curve gives, when evaluated with the least square method using the figures from 0—119 days, a half-life for ^{33}P of 25.36 ± 0.27 days with a confidence level of 95 %. The values from the decay curve with no absorber are rather scattered after about 100 days and a small error in the ^{35}S-correction might interfere with these figures. Therefore the half-life of ^{33}P has been calculated using this curve only from 0—98 days, i.e. about four half-lives. The value thus found was 25.34±0.22 days.

Sample 1, prepared from the 1 kg batch, produced a radiation composed of 87 % ^{33}P and 13 % ^{32}P when using no absorber and 69 % ^{33}P and 31 % ^{32}P when using a 10.1 mg/cm^2 absorber. Fig. 3 shows the decay curves obtained after correcting by means of the ^{60}Co standard and after correcting for ^{32}P. No indications of ^{35}S in the sample could be found. The values obtained for the half-life of ^{33}P were with no absorber 25.19 ± 0.23 days and with the 10.1 mg/cm^2 Al absorber 24.08 ± 0.46 days.

We feel that the results with sample 2 should be given about three times the weight of sample 2. A weighted mean would thus be 25.2 ± 0.2 days. This uncertainty includes counting statistics only. The accuracy of this half-life determination is further dependent on the uncertainty of the half-life of ^{35}S and of the ^{33}P content of the "fresh" sample. The final uncertainty is further dependent on the absorber procedure and might therefore be estimated conservatively to be half a day. Our final result is thus 25.2 ± 0.5 days.

The figure obtained is in accord with Westermark's determination [4] but is not in accord with that of Nichols and Jensen. The uncertainty is however still too high and a new determination would be desirable. Since a ^{35}S enrichment apparatus (based on ion exchange) is in operation here now, we hope to produce millicurie amounts of rather pure ^{33}P and to determine its decay properties, including the half-life, more accurately than has been possible with the rather weak samples studied hitherto.

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