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THE USE OF A RADIOACTIVE TRACER (BROMINE 82) TO STUDY FLOW PATTERNS IN A SUGAR SUBSIDER

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Abstract

The use of radioactive bromine 82 to trace flow patterns in a sugar clarifier is described. The results show that the raw solution goes first to the bottom of the subsider then rises essentially as a layer becoming progressively more diffuse by mixing with solution above and below. The experiment was successful and improvements in technique for future investigations are proposed.

CONTENTS

1. INTRODUCTION

The first stage in the production of sugar is the crushing of the sugar cane and the extraction of its contained juice. Lime is added to the juice to precipitate impurities and to correct the pH to 7 to prevent inversion of sucrose. The juice then passes via a "flash tank" into one or more large settlers or subsiders. One type of subsider (Bach) consists of a large tank containing a series of conical baffle plates or trays and a central spindle on which are mounted arms which rotate very slowly. Chains are attached to these arms to keep the settled mud moving on the trays. The take off points for the purified juice are situated in the apex underneath each baffle. As the arms rotate, the mud and other impurities contained in the sugar juice slide down the baffles to the outside edge of the tank where they sink to the bottom of the subsider and are removed from time to time. The clear juice from each of the six outlets under the baffles is combined in a header tank and then passes on to the next stage of the plant (the evaporators.)

For some time, the Bureau of Sugar Experiment Stations in Brisbane, Queensland, has been investigating the losses of sucrose in subsiders and this has necessarily involved sampling of the juice entering and the juice leaving a subsider. Several methods of determining the residence time have been considered including variation of the pH by the addition of extra quantities of lime. None of the methods considered or tried appeared to be satisfactory and the Isotopes Section of the Australian Atomic Energy Commission was approached regarding the possibility of using a radioactive tracer to obtain more information on the performance of these subsiders. Apart from residence time, the investigation would yield information on the flow patterns in a subsider.

It was decided that it would be feasible to carry out a tracer experiment of this type. A similar experiment in Sweden, [Wramstedt (1)],yielded useful information on the performance of two Dorr subsiders operating on first carbonatation juice in a beet sugar factory. After some negotiation between the Bureau and Millaquin Sugar Company it was decided to carry out the tracer experiment at this Company's Qunaba Mill in Bundaberg, Queensland.

2. DESCRIPTION OF SUBSIDER

The subsider chosen for the test is shown diagrammatically in Fig. 1. It is a 5 tray, 18 ft. Bach subsider of approximately 20,000 gallons capacity. The raw juice, at a temperature of about 215°F, enters the flash tank under conditions of high turbulence. From here the juice enters the subsider through a large diameter pipe, ensuring a low linear velocity of the feed, with consequent reduction in turbulence in the subsider.

During the test the flow through the subsider was about 13,400 gallons per hour, the throughput being measured by a juice weigher. As far as could be determined, the conditions within the subsider were reasonably constant during the test.

3. CHOICE OF ISOTOPE

Because it was desired to use scintillation counters on the outside of the steel outlet pipes from the subsider, a gamma-emitting isotope was required. Also, to ensure rapid self-disposal, an isotope with a comparatively short half-life was required. Three isotopes, iodine 131 (half-life 8 days), bromine 82 (36 hours) and sodium 24 (15 hours) met these requirements.

The first choice was iodine 131, but it was not used because it was not possible to guarantee that the sugar produced in the experiment would be stored for at least 3 months before shipment. The isotope finally chosen was bromine 82, which has a gamma energy sufficiently high to enable detection from outside a steel pipe of wall thickness 1/4 inch, but still somewhat lower than that from sodium 24; hence the effect of "cross-interference", the problem of one scintillation head being affected by radiation coming from somewhere other than the measuring point, is reduced.

Because the A.A.E.C. reactor HIFAR was not yet producing bromine 82, the required amount was imported from England.

4. DESCRIPTION AND CALIBRATION OF EQUIPMENT

Each detecting unit consisted of a scintillation head containing a 1¾ in. diameter x 1 in. length NaI crystal and a photomultiplier. This was attached by 6 to 12 ft. of cable to either an Airmec radiation monitor, type 1021B or an Ericcson Telephones Ltd. monitor, type 145A.

The scintillation heads were fitted into lead collars attached to the outside of the pipes by metal bands at the monitoring points (Figs.2, 3 and 3A.) The lead collars were used to decrease the background radiation and also to minimise cross radiation from the subsider and associated pipe work.

The half thickness of water for bromine 82 gamma radiation is approximately 4 inches; hence if the scintillation head is held against the mid-point of a 2 ft. 6 in. length of pipe, this will present virtually an infinite length of pipe, to the detector.

Five of the six pipes were of 4 in. diameter and the sixth of 6 in. diameter. Thus, the equipment was calibrated in the laboratory by using 3 ft. lengths of 4 in. and 6 in. diameter pipes (covered with a ¾ in. layer of magnesia lagging) and attaching the lead collars to their mid-points as described above. There was some variation between the sensitivities of each detecting instrument. Typical results for the 4 in. and 6 in. diameter pipes are given in Figs. 4 and 5. From the calibrations, it is seen that, with a background of 30 counts per second (as obtained in the laboratory), a concentration of 0.1 microcuries(μ c) per gallon in the 4 in. pipe and 0.05 μ c per gallon in the 6 in. pipe is readily detectable, (i.e., gives about twice the background count).

The effect of cross radiation from other pipes was also checked in the laboratory and found to be negligible at the concentrations used. It was not possible to check in the laboratory the effect of cross radiation from the subsider, but this was minimised in the actual experiment by placing the scintillation heads on the side of the pipes remote from the subsider where they were protected from the subsider radiation by three thicknesses of steel and four inches of juice.

The minimum detectable activity in the 4 in. pipe is 0.1 μ c per gallon and the capacity of the subsider is 20,000 gallons. Hence, assuming complete mixing, the total activity for detection is 2 millicuries. To allow an adequate margin for detection, the actual amount of bromine 82 used in the experiment was 5 millicuries.

Prior to the experiment, the background radiation recorded by the scintillation heads in their lead collars against the pipes of the subsider was surprisingly low -- only 4 counts per second as against 25-30 counts per second under similar conditions in the laboratory. Even allowing for the fact that the laboratory background was slightly higher than normal, due to the proximity of stored sources, the mill background figure was very low. This might have been due to the fact that the subsider was mounted on a steel platform about 23 feet high and was thus well away from any ground radiation. Partly because of this very low background, the 5 millicuries of bromine 82 used in the experiment was more than enough. In any future experiment about one millicurie of activity would be sufficient.

5. DESCRIPTION OF EXPERIMENT

The monitoring points on the subsider are marked X in Fig. 1. During the experiment, one instrument served two monitoring points alternately, i.e., head No. 1 served pipes 1 and 2, head No. 2 pipes 3 and 4 and head No. 3 pipes 5 and 6. Another scintillation head was used to monitor the activity flowing in the pipe between the flash tank and the subsider. This monitoring point is also shown in Fig. 1.

The 5 millicuries of bromine 82 in the form of 7 grams of irradiated ammonium bromide, supplied by the Radiochemical Centre, Amersham, England, was dissolved (using remote handling tools) in 5 gallons of water containing one pound of potassium bromide as a carrier. This solution was contained in a steel tank and after being sampled (see section 6) was transferred rapidly (in about 30 seconds) to the flash tank, by means of compressed air. The experiment started at 9.39 a.m. on Wednesday, 23rd September 1959.

6. SAFETY PRECAUTIONS

Before adding this solution to the flash tank, duplicate samples were sent to the A.A.E.C. Research Establishment at Lucas Heights (Sydney) for radiochemical analysis, as a safety check on the composition of the radioactive material.

The bromine was precipitated as silver bromide, centrifuged and counted (end window Geiger counter) for half-life determination. The supernatant liquid was also checked for activity, but none was found. Another sample of the liquid was counted with a scintillation counter for half-life determination. These tests confirmed that the activity of the sample was due entirely to bromine 82.

The maximum body burden for bromine 82 is 11 microcuries and the amount of this isotope (half-life 36 hours) used in the test was 5 millicuries. This means that in a fortnight, the activity involved would have decreased to less than the body burden. Hence even if all the activity appeared in the sugar, after about 2 weeks one body burden of bromine 82 would be distributed throughout the whole 60 tons of sugar produced in the test. In fact, most of the activity appeared in the molasses which is stored for six to twelve months before processing in the distillery so this was an added safety factor. It was arranged that the sugar produced in the test would be stored in the Bundaberg bulk terminal for three months before shipment, as a further safety factor.

When the active sugar first arrived at the bulk store about one day after the experiment it was monitored and showed an activity of 2–3 times background. This activity became undetectable after about 7–10 days. Radiation readings were also taken at various points around the sugar mill during the experiment, e.g., on mud outlets, maceration, syrup from effets, massecuite, etc. These readings indicated very low, if any, levels of radiation around the plant.

All these facts indicate that, as anticipated, there was no danger at all to any personnel in the mill or elsewhere during or after the experiment.

7. RESULTS

The readings from the scintillation heads were corrected for background and decay (all readings were normalised back to the start of the experiment) and then the readings were converted to microcuries per gallon using the previously prepared calibration curves.

7.1 Activity in Pipe connecting Flash Tank and Subsider

This pipe was monitored by another separate scintillation head, very lightly shielded against the direct radiation from the flash tank and the subsider. For the first few minutes after addition of the bromine 82 to the flash tank, the monitor on the connecting pipe was off scale. This monitor came back on scale after about six minutes.

The contents of the flash tank undergo violent mixing so that the activity added to the flash tank would be completely and rapidly mixed. In this case, the transfer of activity to the subsider would follow an exponential law, i.e.,

$$-\frac{dc}{dt} = \left(\frac{R}{V}\right) \times C$$

where V = volume of flash tank, 715 gallons,

R = flow rate, 13,400 gallons per hour,

and C = activity concentration in the flash tank.

If $t_{\frac{1}{2}}$ is the time taken for the concentration to reach half of the original concentration in the flash tank, then the expression may be reduced to :–

$$\frac{R}{V} = \frac{0.693}{t\frac{1}{2}}$$

$$\therefore \quad t\tfrac{1}{2} = \frac{0.693 \times 715}{13{,}400} \times 60 \text{ minutes}$$

$$= 2.2 \text{ minutes,}$$

i.e., the time to transfer half the activity to the subsider is 2.2 minutes.

Figure 6 is a graph of the activity–time curve for this connecting pipe. From the early part of this curve, the "half time" of transfer is 2.9 minutes, which is in reasonable agreement with the theoretical value. There is a sharp change in the slope of the curve about 15 minutes after the start of the experiment. This is probably due to the fact that the first pulse of added activity had now gone around the subsider once (the addition is tangential) and was then affecting the monitor by cross radiation from the subsider (this monitor was virtually unshielded.) This means that the initially added material rotates in the thickener at about 4 rev. per hour. The subsider blades rotate at about 6 rev. per hour, but it is thought that the circulation of the juice would not be influenced to any extent by the subsider blades which are provided merely to keep the settled mud moving on the trays. The juice circulation would be imposed by the velocity of entry and in any case, a feed trough is provided around the subsider by which, perhaps, uniform feeding of the unit is achieved.

7.2 Activities at Subsider Outlets

The subsider has six outlet pipes, numbered consecutively from 1 to 6, starting from the bottom of the subsider. After converting the activity readings (counts per sec.) to microcuries per gallon, the values were plotted against time, for each pipe (Figs.7 to 9.) The reading obtained is, of course, independent of flow velocity in the pipe, provided the mixing is uniform.

The high readings recorded during the first 5 or 10 minutes are probably due to direct radiation from the flash tank, before a considerable portion of activity had left it. This is supported by the fact that the nearer the particular monitor is to the flash tank, the higher is its initial reading, e.g., for pipe No. 1 (closest) initial reading, 1.4 μc per gallon and for pipe No. 6 (furthest away), initial reading 0.5 μc per gallon.

It is possible that this cross interference effect lasts longer than a few minutes, but the relative effect would become smaller as the experiment proceeded. However, because of this cross interference, it is difficult to tell when the activity first appeared at each outlet.

After the first five or ten minutes, the readings are probably realistic. They show clearly that the activity goes to the bottom of the subsider first, then flows upward from there, becoming more dilute as it rises. The respective values are:–

Pipe	1 (bottom),	max.	reading	1.25	μc	per	gallon	at	10	minutes.
"	2	"	"	1.04	"	"	"	"	17	"
"	3	"	"	0.97	"	"	"	"	22	"
"	4	"	"	0.86	"	"	"	"	31	"
"	5	"	"	0.78	"	"	"	"	46	"
"	6	"	"	0.10	"	"	"	"	60	"

From the shapes of the curves and their relative positions it is concluded that the activity (and hence the juice) is passing upwards through the subsider substantially as one layer with mixing becoming more and more pronounced as the juice nears the top of the subsider.

The total flow rate through the subsider during the experiment was 13,410 gallons per hour and the flow through each of the individual outlets was substantially the same at 2,235 gallons per hour.

By integrating the area under each of the curves, the amount of activity passing through each outlet in two hours was found. This applies, irrespective of the shape of the individual curve. These values are:-

Curve 1	1.81	millicuries
2	1.62	”
3	1.45	”
4	1.40	”
5	1.20	”
6	0.37	”
Total	7.85	”

The total is more than the added amount of bromine 82 (5 millicuries). The reason for this could be either interference from the subsider, or, more likely, a difference between the calibration of the small amount of bromine 82 produced at Lucas Heights for calibrating the equipment and the bromine 82, ex Amersham, used in the experiment.

Because the volume flow through each outlet pipe was the same (special efforts were made before the experiment to ensure this) the total activity passing through each outlet should have been the same. However, as can be seen from the above, the activities obtained for the first two hours were substantially different for each outlet. The reason for this is not clear; Wramstedt, in his paper on the Swedish test (loc. cit.), encountered the same effect, the relative areas being:-

Upper outlet	124	units
Middle ”	86	”
Lower ”	103	”

From the activity curves, it is clear that most of the added feed material had passed through the subsider after about two hours. However, it was not until about seven hours after the start of the experiment that the activity readings at the outlets returned substantially to background. This indicates that a small amount of added material undergoes very great dilution.

The results in general indicate that the distribution of juice in the subsider is quite unexpected. Juice flows from the circumferential trough down through triangular feeders, on the side of which slots are provided. These slots are provided to achieve some distribution on to the trays. At Qunaba, either these slots are of insufficient size or they were blocked to some unknown extent at the time of the experiment. It might be worthwhile enlarging these slots for better distribution of juice.

In regard to the residence time of juice in such a subsider, it is evident that it would be difficult to obtain corresponding samples of inlet and outlet juices, due to the mixing already mentioned.

7.3 Suggestions for Future Experiments

The following suggestions are made for any future experiments:-

(a) Cross interference could be reduced by using more efficient shielding on the scintillation counters and/or by using a lower energy gamma-emitting isotope (e.g., iodine 131).

(b) A smaller amount of activity (about 1 millicurie) could be used.

(c) It might be better to add the tracer to the subsider in the form of a pulse instead of exponentially via the flash tank, as in this method.

(d) The combined clear juice outlet pipe could be monitored, well away from the subsider, to eliminate interference from cross radiation. This would give more reliable information on the initial appearance of activity emerging from the subsider (hold-up time) and a check on the total activity emerging from the subsider.

8. CONCLUSIONS

Summarising the results of the experiment, we find that:-

(a) The raw feed material entering the subsider goes firstly to the bottom of the subsider and works its way up from there.

(b) The liquid moves up through the subsider essentially in layers with mixing becoming more and more severe. The extent of mixing can be determined by the shape of the individual outlet curves.

(c) Most of the feed material has passed through the subsider in two hours.

(d) The juice distribution was not that normally envisaged and some attention might be paid to this aspect of the subsider operation.

(e) As mixing in the unit is substantial, it is doubtful whether any reliance could be put on the corresponding juice samples taken at the inlet and at the outlet of the unit. This position, perhaps, only applied at the crushing rate encountered during the experiment. Conditions could be more favourable at other rates.

(f) The experiment proved the feasibility of using radioisotopes for investigations in sugar mills with absolute safety, provided the work is carefully planned and carried out by responsible personnel. The fact that radioisotopes are now available from the A.A.E.C. will make their use more attractive in the future.

9. ACKNOWLEDGMENTS

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Our thanks are due to the Commonwealth X-Ray and Radium Laboratory for their co-operation in expediting the delivery of the bromine 82 from England.

The ready co-operation of the Bulk Sugar Handling Committee, the Colonial Sugar Refining Company and the Departments of Health in Queensland and Victoria is gratefully acknowledged. Our special appreciation is due to the management of the Millaquin Sugar Company and the management and staff of Qunaba Mill, without whose co-operation this experiment would not have been possible.

REFERENCES

1. Wramstedt, S. 1958. Socker Handlingar II, 14 No. 6, p. 113.

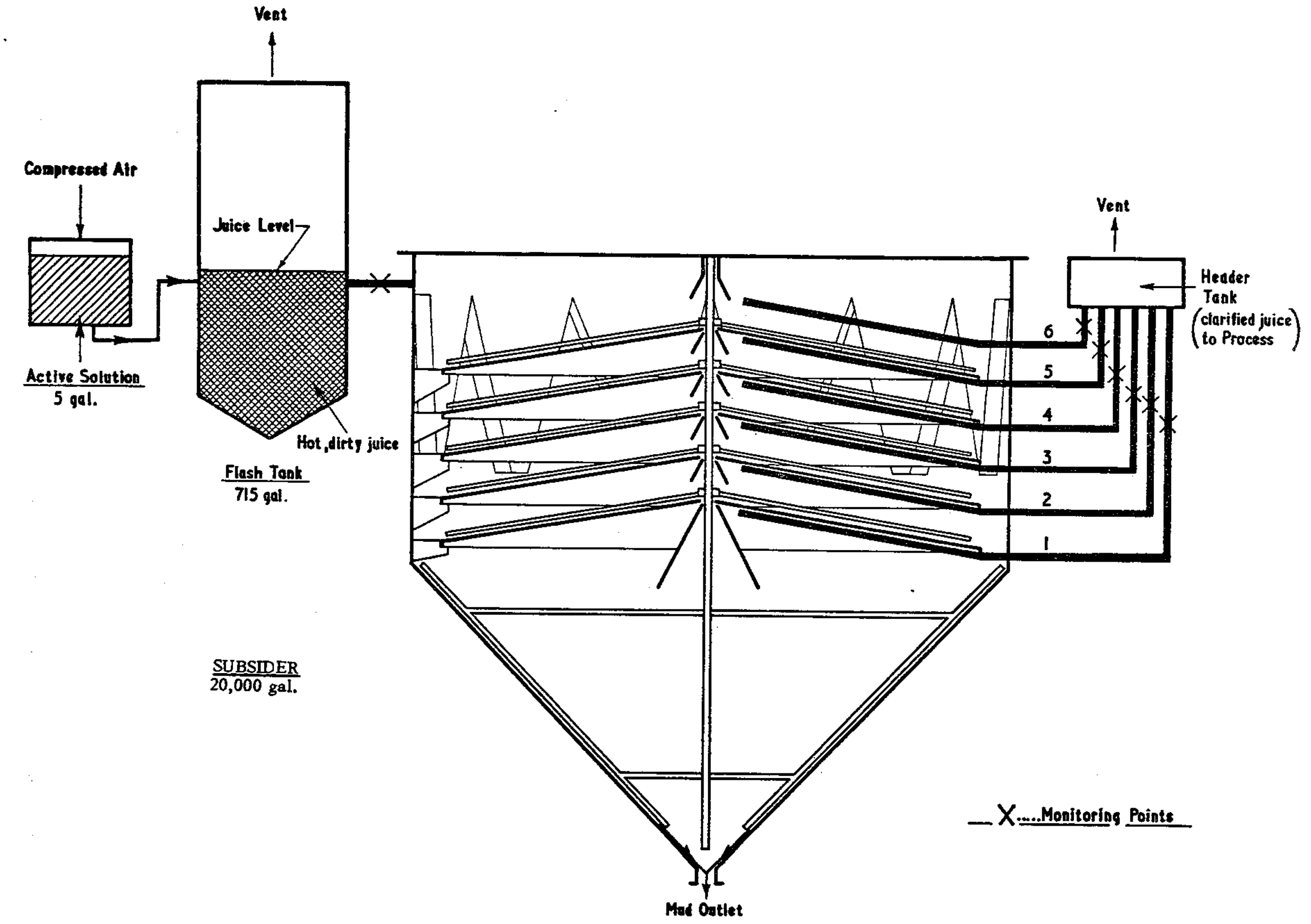

FIG. 1 – SUBSIDER INSTALLATION, SHOWING MONITORING POINTS AND METHOD OF ADDITION OF ACTIVE SOLUTION.

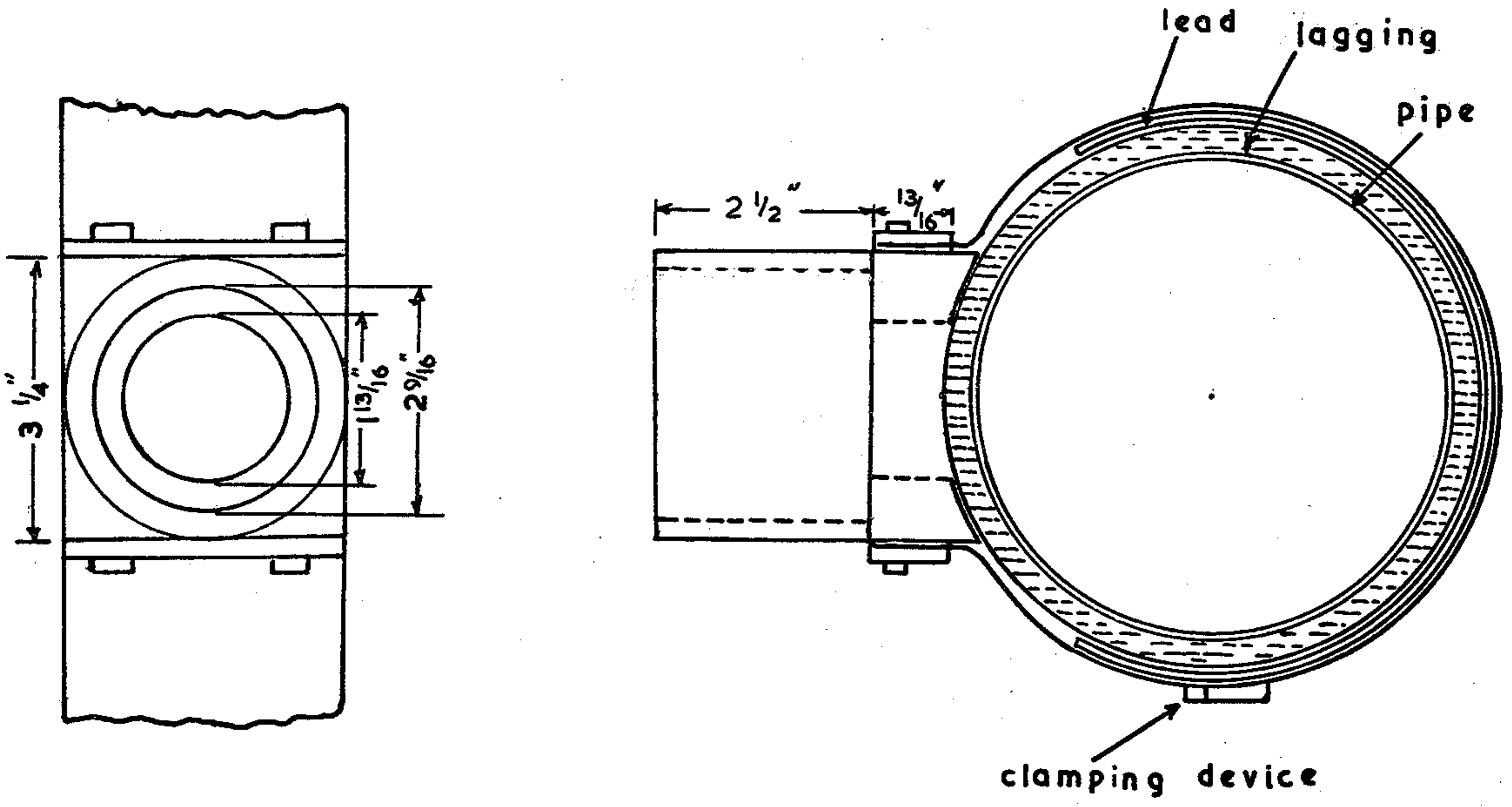

FIG. 2 Lead collar for Scintillation Head

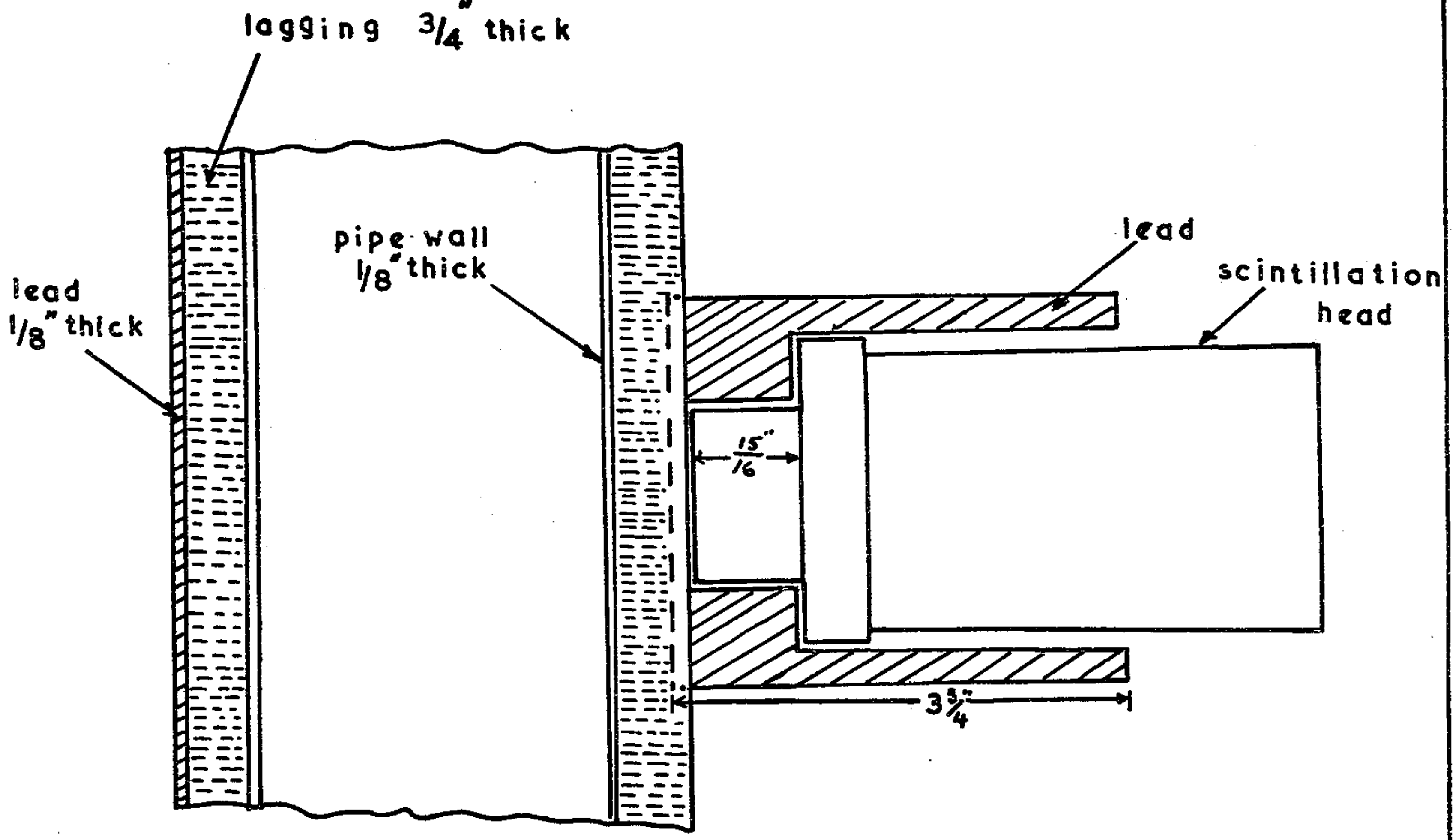

FIG.3 Scintillation head in lead collar attached to pipe

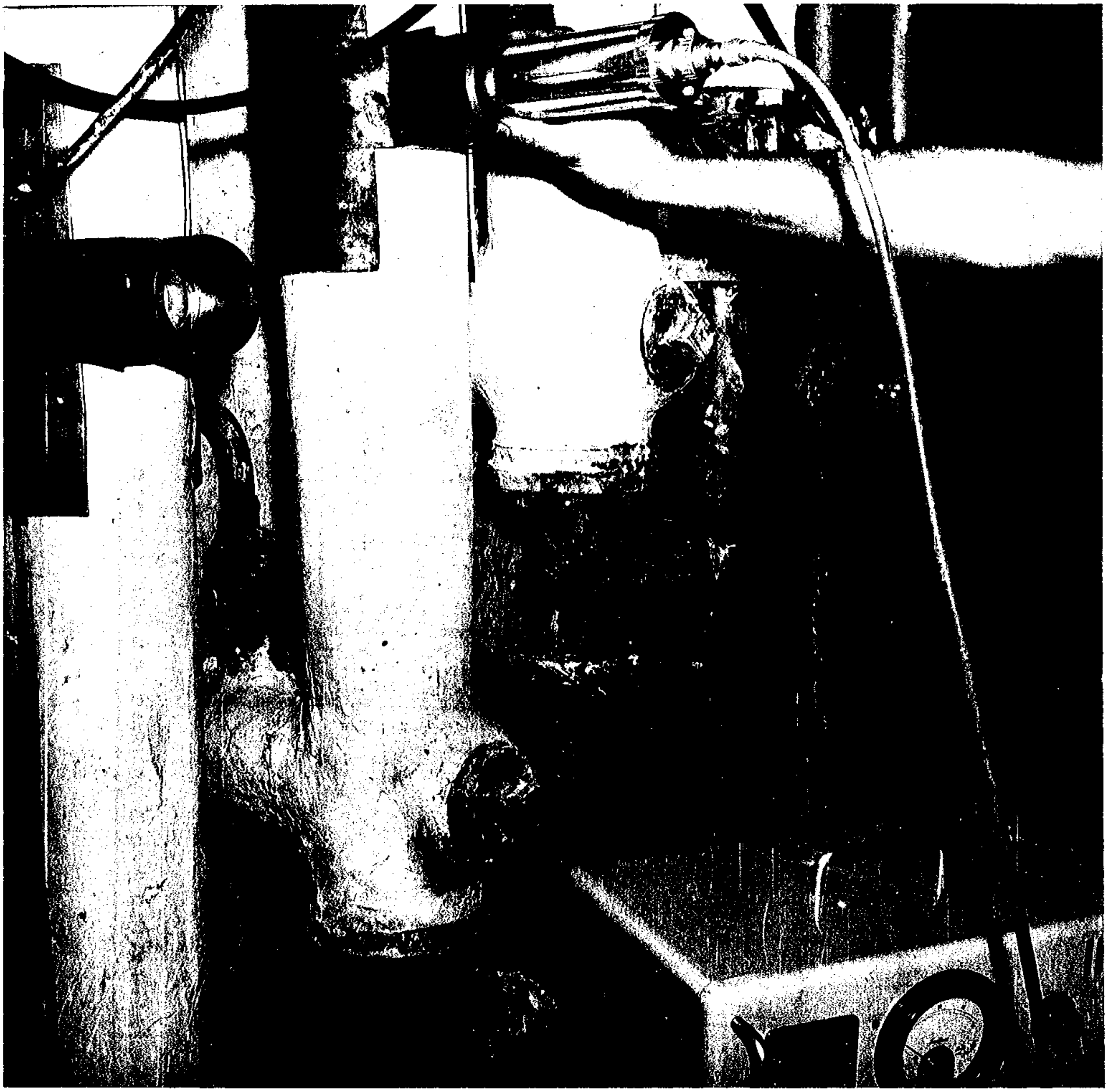

FIG. 3A. SCINTILLATION DETECTOR HEAD IN LEAD COLLAR AT MONITORING POINT.

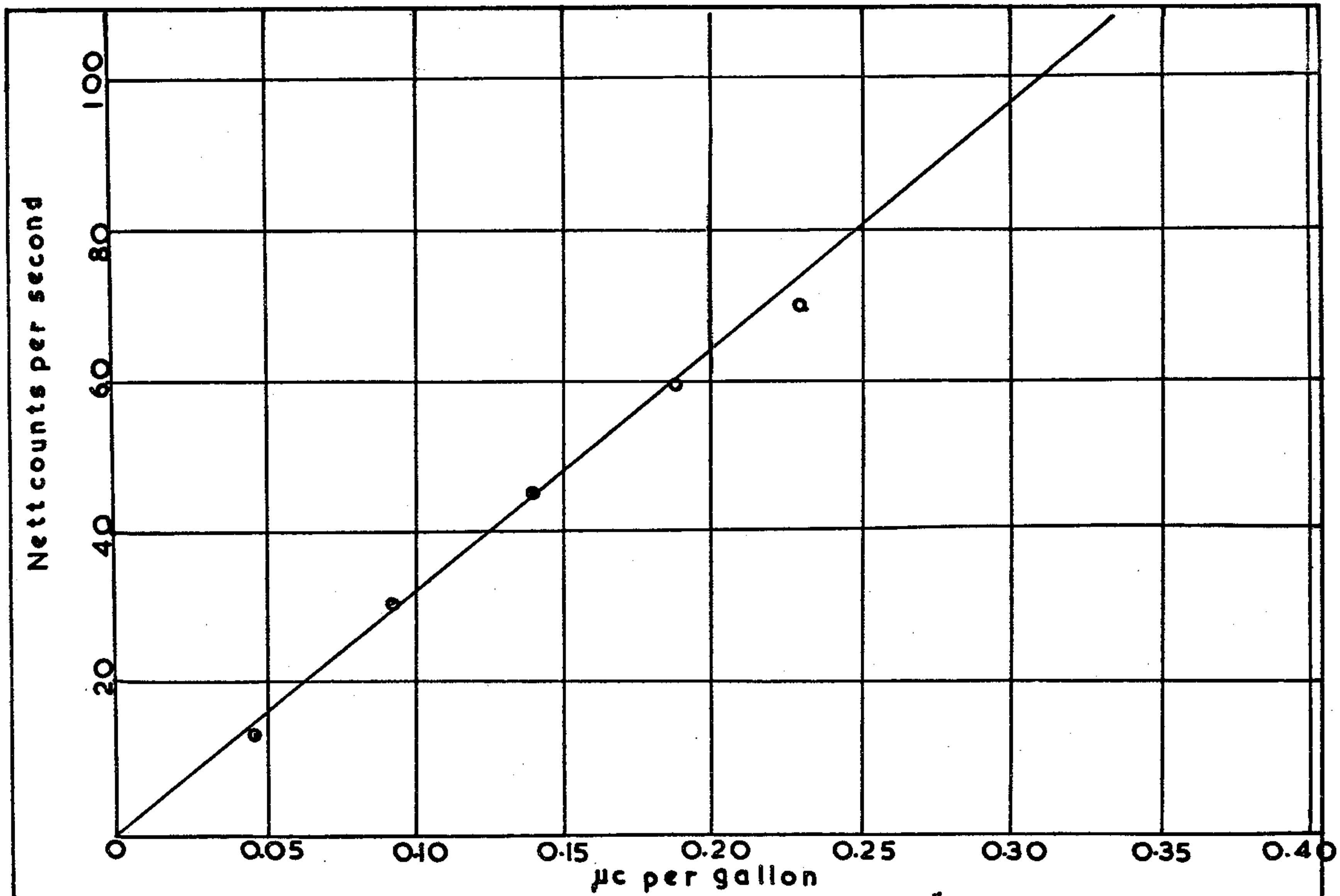

FIG 4 Calibration of equipment 4" diam. pipe

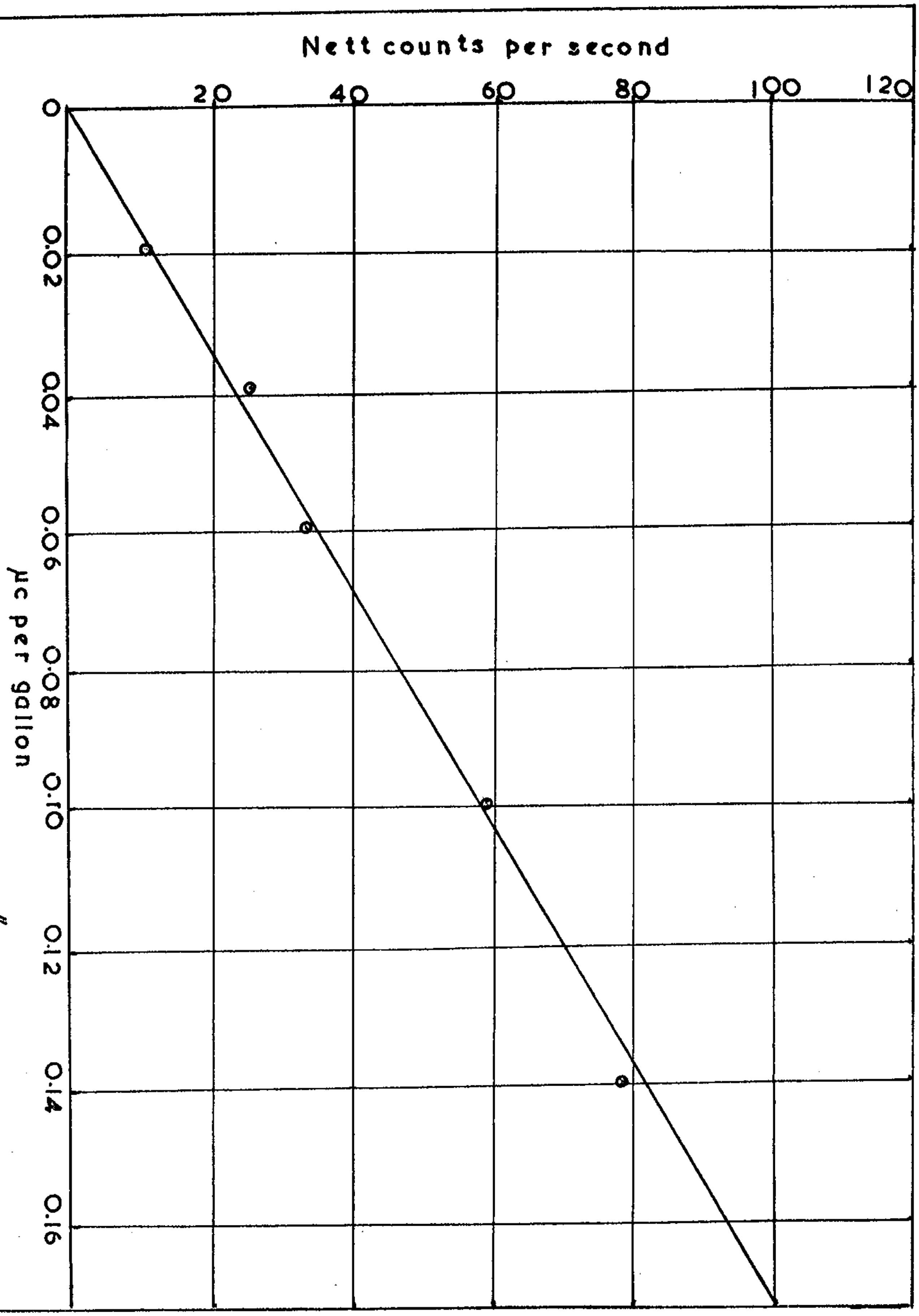

FIG. 5

Calibration of equipment 6" diam. pipe

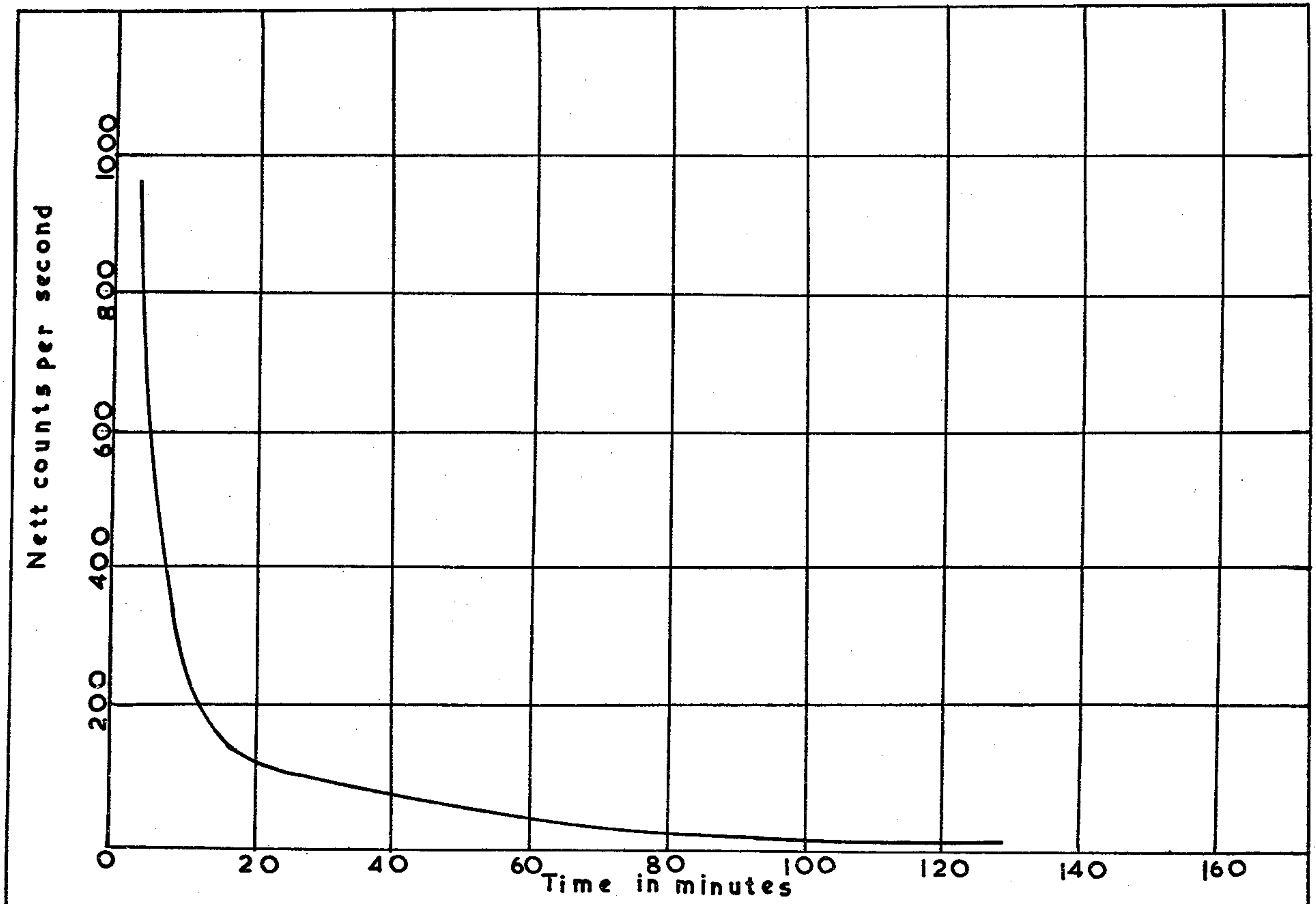

FIG. 6 Activity in pipe connecting flash tank and subsider

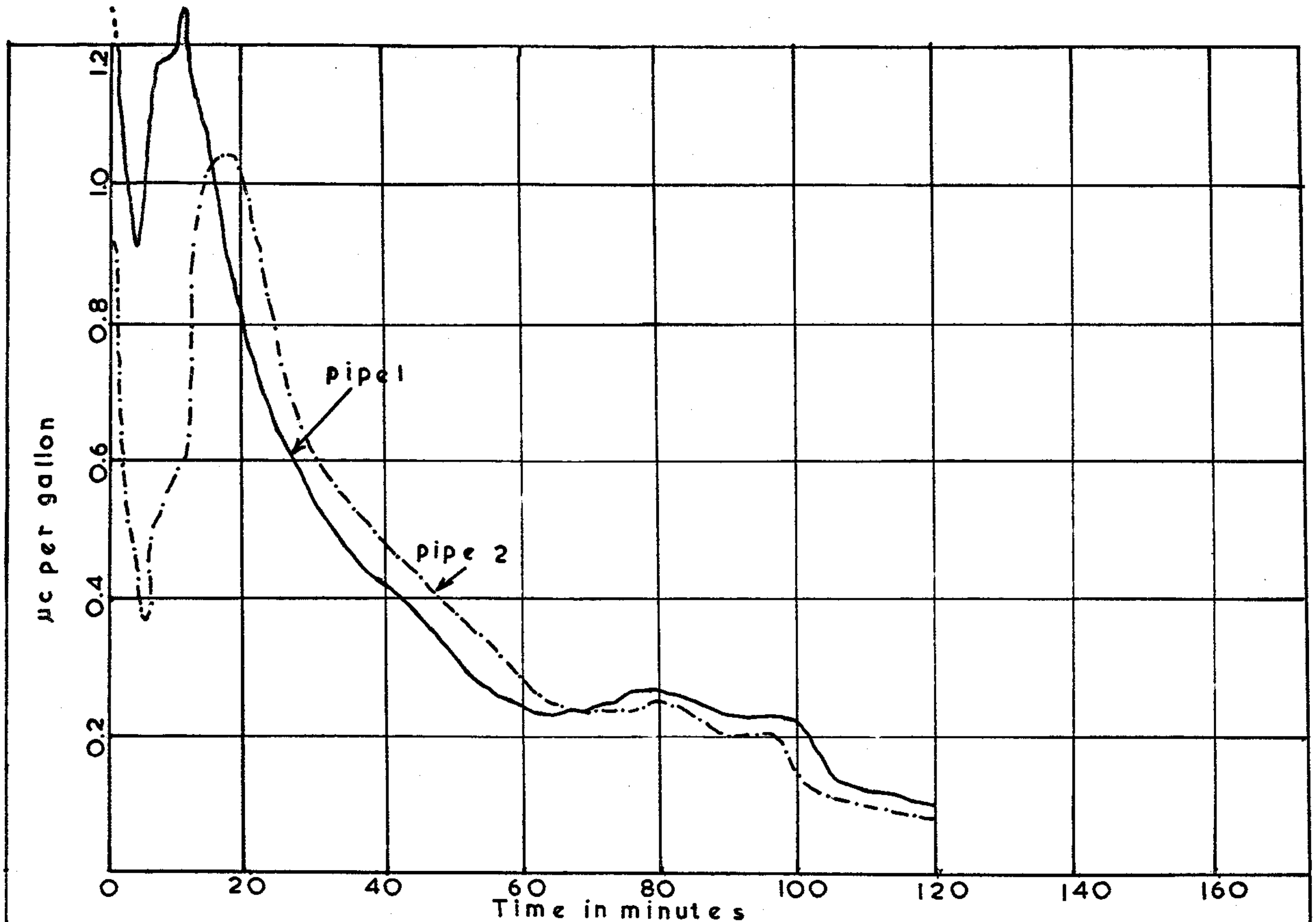

FIG.7 Activity in pipes 1 and 2

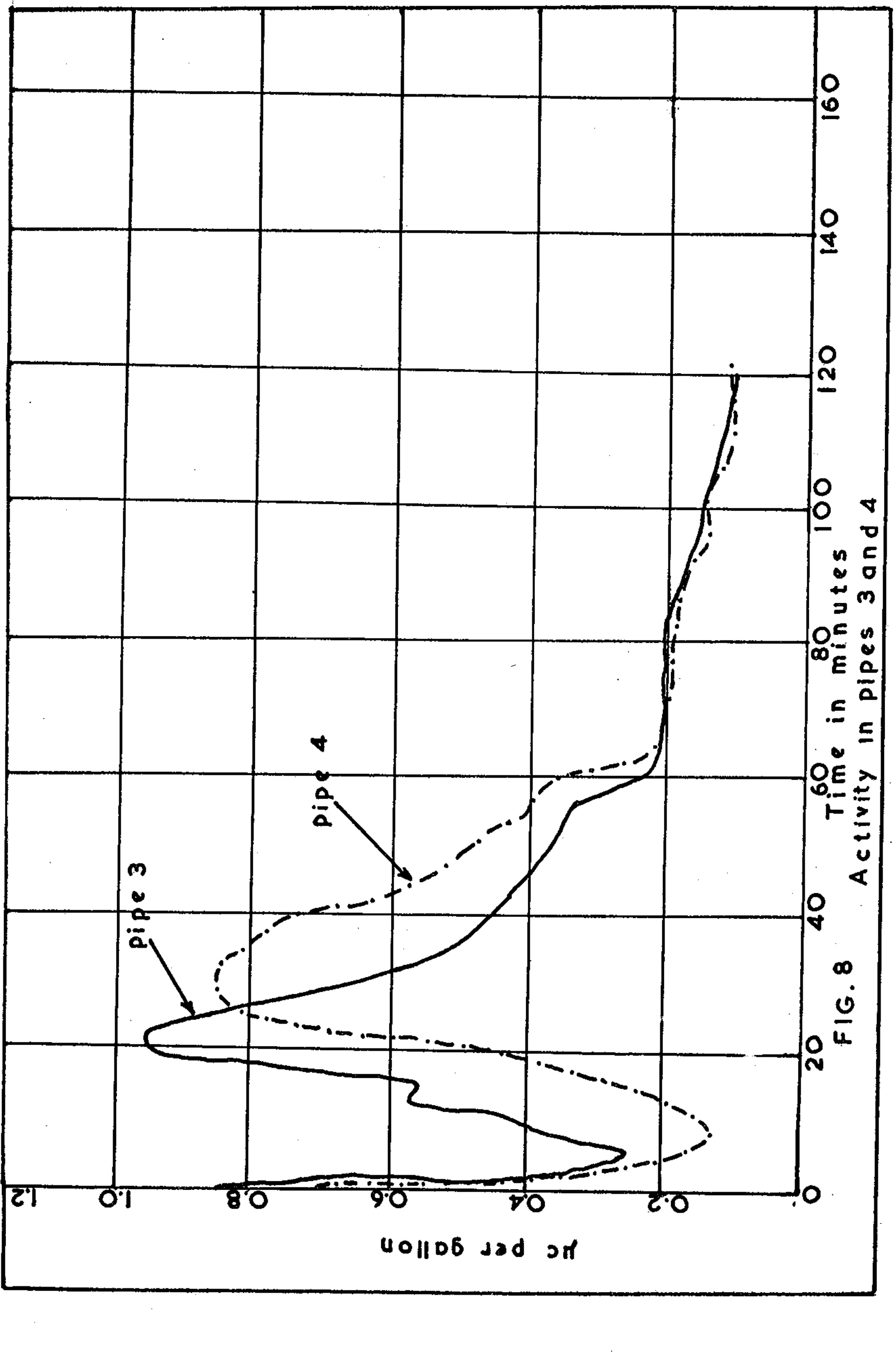

FIG. 8

Activity in pipes 3 and 4

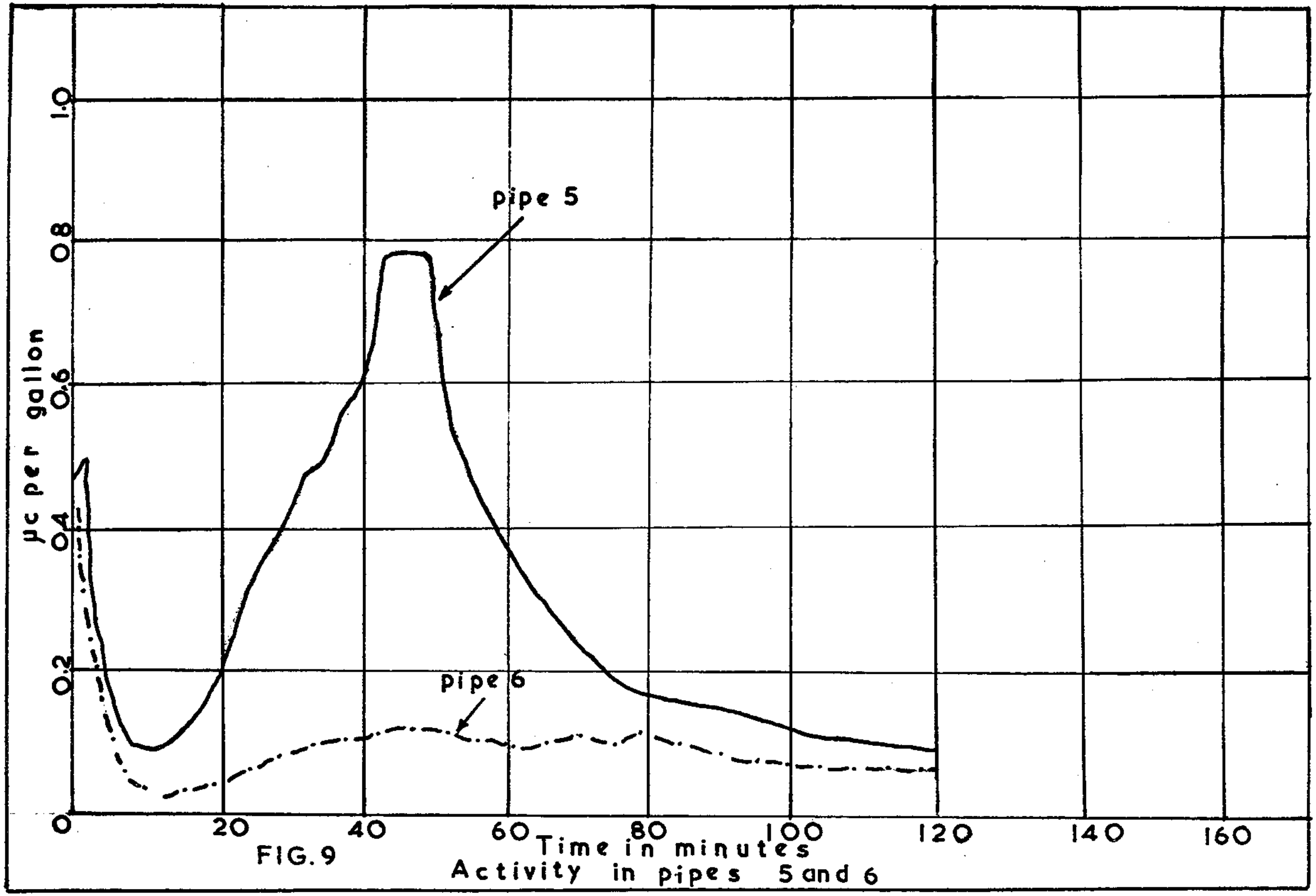

FIG. 9
Activity in pipes 5 and 6