The use of X-ray for quantifying lead shot pellets in substrate samples

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Lead poisoning of waterfowl, following ingestion of lead shot from shotgun cartridges, has been shown to be an important mortality factor for some species. In North America, Bellrose's (1959) study suggested there was an increasing incidence of lead shot contamination from grazers (e.g. geese and swans), through dabbling ducks to diving ducks.

In the U.K. the problem of lead shot contamination of the environment would appear to be less than it is in many other parts of Europe and America, although data are sparse and there is considerable variation between sites. For example, lead pellet densities of $1-44/m^2$ (10,000–440,000/ha) were common in the U.S.A. (Thomas 1982) and 8 out of 19 sites examined had densities in excess of 300,000/ha (Moore and King 1979). Also Bellrose (1959) reported up to 291,700 pellets/ha in N. America. In Denmark, concentrations of up to 4,000,000/ha were found in areas where active clay pigeon-shooting occurred (Petersen and Meltofte 1979). In the U.K., Mudge (1984) found a range of densities up to $30/m^2$ (equivalent to 300,000/ha) but 3 out of 22 sites had no pellets at all.

There are many problems associated with locating and quantifying lead pellets in the environment and most workers have used a sampling procedure followed by sieving and searching (e.g. Mudge 1983, 1984). Depending upon the size of samples taken, and the particle sizes of the substrate material, the process of searching samples by eye can be very labour intensive and thus expensive. In addition, there is the difficulty of determining how efficient the searching has been, and hence the accuracy of the results.

During the course of a study of lead poisoning in Whooper Swans, the possibility was examined of using radiography as a means of detecting and quantifying lead pellets in sieved substrate samples and in waterfowl gizzard grit. (Fredrickson *et al.* 1977; Anderson and Brewer 1980).

Samples were taken from the intertidal zone of the Ythan estuary, using a corer of 70 mm diameter ($38.48 cm^2$) to depths varying from 100–200 mm. The samples were sieved through a 1 mm mesh, and the material retained was hand-sorted to remove all stones greater than about 15 mm across. The rest of the material was then checked by eye for the presence of lead pellets. Each sample was searched until the examiner was satisfied that no more pellets could be found within a reasonable time period. Search times varied according to the amount and nature of material, with a mean of 9 minutes and a range of 5–20 minutes for each sample.

For the radiography a Hewlett Packard Faxitron 43805N X-ray machine was used to record the images on Kodak Industrex 620 paper, held for the purpose of exposure inside black double PVC wallets.

The samples, each individually packed in polythene bags, and accompanied by an X-ray opaque lead foil label, were placed in the loaded PVC wallets inside the machine and were flattened to about 5 mm thickness. To improve definition, no intensifying screens were used which necessitated long exposure times of usually between 1 and 2 minutes for most soil/mud samples. The X-ray machine settings were 50kV at 3mA with a source to subject distance of about 60 cms. After exposure the images received the normal photographic print development procedure for 'fibre based' papers.

As a rough guide, 3 to 4 samples and their labels could be recorded together on the 18 x 24 cm size industrex paper with an approximate material cost of 50p per sheet. Thirty samples were treated in this way, and two of them were repeated giving a total of 32 for comparison (Table 1). A total of 18 pellets were found by eye, whereas 43 were recorded in the radiographs. Where there were pellets present in the samples, the observer found the 'correct' number on only 4 occasions. On one occasion the observer recorded two pellets, although only one was found on the X-ray plate. In a

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subsequent re-examination of the samples, the mean search time required to locate the number of pellets revealed by radiography was 15 minutes, with a range of 9 to 25 minutes.

To test the accuracy of the techniques, known quantities of No. 6 lead shot were added to whole unsieved mud samples. In 4 trials all the lead pellets initially added were detected on the radiographs. When searched by hand later, these pellets proved to be the same pellets that were added at the beginning. Thus one can be confident that no lead shot was lost in the sieving process and all lead pellets were detected by the X-rays.

Table 1. Comparison of the numbers of lead pellets found by eye and X-ray method.

Sample No.	Time to Search (mins)	No. of Pellets Found	
		By Eye	By X-ray
A4	4	2	3
A5	10	0	0
A17	12	1	3
A17	9	1	3
A22	8	1	1
A26	6	1	2
A37	5	2	4
A45	7	2	3
A72	6	0	0
A73	4	0	1
A74	11	3	3
A75	5	0	0
A76	7	0	0
A77	13	0	0
A78	10	0	1
A79	11	0	1
A80	11	0	0
A81	17	0	0
A82	14	0	2
A83	11	0	1
A84	12	0	0
A85	10	0	0
A86	13	0	2
A87	13	0	1
A87	10	1	1
A96	8	0	2
L4	12	0	2
L6	8	0	2
R2	11	0	1
R5	6	0	1
R10	11	2	2
A10	3	2	1
Totals:			
32		18	43

The second test was to ascertain if lead could perhaps be shielded by stones in the samples. Lead shot was fixed with adhesive tape beneath pebbles measuring 20–30 mm in diameter, i.e. of diameter normally excluded by hand sorting. As can be seen from Figure 1 the pellet still shows up very clearly.

Whole, unsieved soil/mud samples were also X-rayed. Lead pellets were detectable (seeding trials were also done on these samples) but the samples required about 5 minutes of X-rays and 1 sample requires a whole sheet of paper (i.e. 5 times as long at 3 to 4 times the cost).

Gizzard grit samples were also X-rayed. These were placed in Petri-dishes and received only 30 seconds of X-rayed exposure. The rest of the procedure was identical.

One can be confident, after re-checking to find the extra pellets, that the X-ray technique is 100% efficient and that it has demonstrated a source of error in the previously widely accepted method of searching by eye. Most of the pellets found in the sediment were discoloured, etched, mis-shapen, or had a surface coating of some organic material, all of which caused them to become highly cryptic among the sand grains. Any additions of fresh new pellets to the samples could quickly be seen, since they were dark in colour and nicely rounded in shape. It was concluded that previous attempts at testing the searching efficiency of observers may have been in error, if fresh new lead shot had been used in the seeding trials.

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Summary

X-radiography was used to detect lead pellets in intertidal mud samples. The samples, removed by a corer, were sieved and searched by eye for lead, with a mean search time of 9 minutes (range 5–20 minutes). A total of 18 pellets were found in 32 samples. The samples were then placed

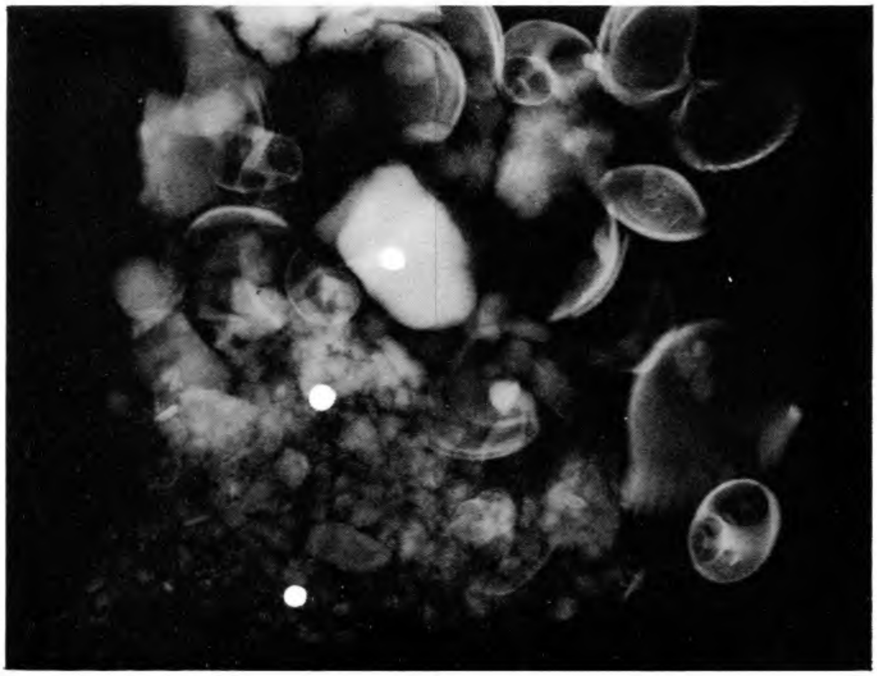

Figure 1. X-ray photograph of substrate sample showing three lead pellets of No. 6 shot.

upon X-ray sensitive paper and X-rayed for 1–2 minutes. Forty-three pellets were recorded on the radiographs. Subsequently the samples were re-checked and all 43 pellets were found. Mean search time required was 15 minutes (range 9–25 minutes). The technique indicates a large potential source of error in conventional methods used to find lead in mud samples.

Unsieved mud samples and pellets fixed beneath stones were also X-rayed. In all cases the pellets were detectable. Gizzard grit samples were also analysed in this way.

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