SUMMARY AND CONCLUSIONS

A further investigation of the technical fundamental of the method of the tagging of oil residues for identification purposes has been carried out, directed by an appointed project group. The investigation has been concentrated on the basic features of the method and has not included any field experiments.

The results of the work confirm so far that the method of tagging oil residues with metal particles is feasible. It is possible to obtain analytical results to identify the particles with an accuracy which the group considers to be acceptable for the purpose of the method. However, the group deems it necessary that the specifications of the method are made more stringent and also that they are changed in some aspects.

However, only a Dart of the method has been scrutinized. Various questions relating to the means for distributing the tagging material into the oil residues, the actual behaviour of the tagging material in oil slicks and the means for collecting an adequate amount of tagging material from an oil slick 'have not been evaluated in this study. It would be advisable to perform further studies, which should include field experiments, before a complete evaluation of the feasibility of the method is made.

The investigations have shown that more accurately specified tagging material, both with regard to choice of Component and manufacturing procedure will increase the accuracy of the microprobe analytic results and deerease the span between the results obtained when analysing the surfaces of the individual Darticles.

Irregularities noted in particles in the original tagging material may account for some of the deviating results obtained in the previous field experiments.

There is a need for periodical calibration of the analyzing equipment by means of reference samples distributed to appointed laboratories in order to account for effects caused by the characteristics of the tagging material and other factors pertinent to the analyzing technique being used.

The tests have also shown that personnel must be highly qualified and that laboratory equipment must be of a high standard. Together with the need for standardized calibrations mentioned above, laboratories will have to meet certain approved standards.

The group has identified certain problem areas and has come to the following conclusions.

1. The quality and manufacturing technique of the tagging material influences the accuracy of the analytical results. Certain components should be avoided in the alloys. Two or three well-chosen main components combined with smaller amounts of other suitable metals could increase both the identification possibilities and the number of combinations in such a way that it might to some extent be possible to use only one alloy when tagging oil residues.
2. Light ingredients like silicon give rise to greater errors due to high background signals in the instruments at the low end of the radiation spectrum and should not be used. Easily oxidizable ingredients, such as chromium and presumably manganese, should be avoided if the production of the tagging material is performed in a non-inert atmosphere.
3. The analyses have shown that particles of a given composition can be identified with high accuracy. The standard deviation in the analyses of a relatively large number of particles from one source is in the magnitude of 1%. This is valid for all laboratories which have been involved in the work.
4. In some cases, however, there is a relatively large span between the mean value of the content of the ingredients in an alloy as obtained by different laboratories. The reason for these Variations has not been fully identified but seems to be related to the use of different analysing equipment and difference in or lack of calibration standards. The result may also be influenced by the fact that, in addition to the original laboratory manual of the tagging method, only limited information or instructions were given to these laboratories about what could be expected to be found in the oil. The reason for this was to try to establish the amount of knowledge which is necessary for a laboratory to possess in order to obtain acceptable results. They were thus less familiar with the technique than any laboratory would be which had been specially selected to do the analyses in an operative tagging system. One laboratory failed to identify some of the tagging material and reported findings about other tagging alloys - which seems to indicate a mix-up in the handling of samples or inadequate information at some stage of the work.
5. It seems obvious from the work carried out that only highly qualified metallurgical laboratories should be used for the analysis of tagging material and that training and experience in work with tagging material is necessary for a consistent result. Fully conclusive analyses can only be obtained if specially appointed laboratories, with access to standard reference tagging materials for calibration, are used.

Work has previously been carried out in Sweden during the period 1972 - 1980 and, during some phases, in cooperation between the Baltic Sea States aiming at the development of a tagging technique for oily residues on board ships. The development of this technique, as it was at the end of the joint field experiment carried out by the Baltic Sea States in 1979, was reported in: "Final Report of the Steering Committee for the Joint Field Experiment within the Baltic Sea Area in Tagging of Oil Residues in Tankers", Stockholm, June 1980.

As a basis for further evaluation of the technique it has been considered desirable to reconfirm the basic performance and characteristics of the tagging material.

From a program for the evaluation and verification of the tagging method (AIB, 1981-10-08, appendix 3) the most fundamental questions relating to the reliability of the tagging method have been studied in depth.

The aim of these investigations were

- to show systematically any faults and inaccuracies occurring in the use of the microprobe technique directly on the surface of the single metal particle of the tagging material;
- to compare the results obtained from different laboratories in analysing tagging material extracted from prepared tagged oil samples;

- to make at least preliminary investigations of the behaviour and redistribution possibilities of tagging material stored in diesel oil for long times;
- to collect and describe experience gained from practical laboratory work in isolating tagging material from the oil samples as well as making the microprobe technique as efficient and accurate as possible.

Together with these tasks it has been necessary, to some extent at least, to specify desirable properties of the tagging material in order to avoid unnecessary analytical inaccuracy caused by the surface irregularities of the tagging material.

This report covers laboratory work carried out on this subject in Sweden during the period July - October 1982. The laboratory work has been explicitly directed on the following questions:

1. Verification of properties of the tagging material, i.e. size, shape, composition, homogeneity.
2. Verification of accuracy and repeatability of the analyses.
3. Verification of the possibilities of isolating and identifying tagging particles in oil residues.
4. Verification of the behaviour of the tagging material suspended in diesel oil, i.e. in prepared tagging solutions.

Some variations in the result of the analyses were noted and traced to lack of homogeneity on the surface of the particles caused by the formation of oxides. The investigation was therefore extended also to cover metal particles also from two other sources. These other particles are, in contrast to the original tagging material, produced in an inert atmosphere and, therefore, contain only very small amounts of impurities.

The laboratory work has been carried out by the Swedish Institute for Metals Research in Stockholm and analyses of particles suspended in oil residues have been carried out by three other laboratories.