ELECTRICITE DE FRANCE
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GROUPE DES LABORATOIRES

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ROBOTIZED DISSOLVING SYSTEM

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The chemical laboratory, part of EDF Hot Laboratory (SCMI), has bought a ZYMATE robot (ZYMARK Company) to develop an automatic dissolving system . The topic of this presentation is to point out the purpose of this automatisation, to describe the installation and to explain how it works.

1 PURPOSE OF THE AUTOMATISATION

Our main purpose is to determine the chemical composition of highly irradiated materials (nuclear fuels excluded).This necessitates to dissolve samples in an acid mixture. The liquid solution is then analysed by Atomic Emission Spectrometry in an Inductively Coupled Plasma (ICP-AES), or by Atomic Absorption Spectrometry (AAS).

Robotization of the dissolution process will enhance the analytical possibilities concerning radioactivity :

- analysis on materials with higher radioactivity will be performed without increasing doses intregrated by chemistry lab operators,
- dose reduction estimated to a factor of two, or even more, for preparation work, analysis and effluent treatment.

Let us consider, an analysis on a sample with a dose rate of 0.44 Gy/h (44 Rad/h) at the contact of the sample. The dose balance of this operation (8 individual analyses) carried out manually with usual care has been 2 mSv (200 mrem) involving two operators. This system would enable to reduce this balance to 1 mSv (100 mrem).

The maximum gamma activity permitted in this installation will be 1 Ci as ^{60}Co. This would enable to analyse samples weighing 0.5 g with 75 mCi as ^{60}Co. It has been estimated that the Laboratory will have about 10 analyses to perform each year.

This automatic system would permit a high rate of analysis, but it is not the goal of this development. This "device" replaces traditional laboratory methods which excluded analysis on very irradiated materials or lead to a too important dosimetry for operators. With the use of Zymate, the dissolutions necessary for determination of the chemical composition can be carried out in almost all the cases regarding irradiated materials.

2 TECHNICAL CHOICES

The chemistry lab had two possible solutions to deal with the increase of gamma activity levels on sample to be analysed.

1 Use of lead cells and remote manipulator arms ; this solution is not adapted, considering the difficulty of remote handling acid solution. Furthermore it is an expensive solution.

2 Move the operators far away from the samples. Robotization is very well suited to this goal : operations are repetitive, rapid and carried out within an accuracy of 1 mm. This solution is less expensive than the first one.

3 CONFIGURATION OF THE SYSTEM

This system is located in a dedicated local of the chemistry laboratory. A first zone for the operators has been isolated from the installation by a glazed wall. This area contains the loading air lock for lead casks and a computer for the system monitoring (appendix 2). A second zone containing the Zymate robot is under ventilation. On a wall of this room, an exit air lock enables the specimen transfert to the ICP Spectrometer. Moreover, this room is equipped with a fume chamber, with inside the heating device for acid attacks.

The center part of the robot consists of a motorized arm, with three independent movements : two translations (X, Z) and a rotation. The arm perfom all the handling operation on the specimen and acid solutions. In our case, a translation motion has been added to take the robot away from the chemical agressive area (acid attack) as soon as it's no longer necessary. 17 workstations are situated along a circular line (appendix 1, 3 and 4).

4 PROCESS DESCRIPTION

4.1 DISSOLUTION

First step :
Samples to be analysed come as chips in a lead cask. The conditionning which is used in this case is fitted to the exit air lock of the hot cell and to the transport carriers.

Second step :
Using grips, containers are manually put on a lorry. A pneumatic system introduces the lorry in the working area of the robot [A].

Third step :
Using the monitoring computer, the operator enters :
- the sample name,
- the location of the container to be taken,
- the number of tests,
- the weights of specimens to be analysed,
- the final concentration of the sample,
- nature and percentage of the acid to be used.

NB :
If the weight of the sample is slightly different from the aim value, the robot adjusts the amount of acid and water to fit the acidity and the final concentration which have been previously chosen.

Fourth step :
The robot takes the container and opens it when arrived in the dedicated workstation [R].

Fifth step :
In the case of fine powders, sampling is automatically performed, by means of a "vibrating" hand, directly in the acid attack flask made of teflon (MATRAS), and weighed with accuracy [B] or [E].
In the case of massive chips, sampling is performed by skilled operators with a remote handling grip hand.

Sixth step :
The container is reclosed and put in the lead shelter [D].

Seventh step :
The robot transfers the teflon flask to the workstation [H] where the acids (chlorhydric and nitric) are added.

Eighth step :
In order to obtain a good dissolution the teflon flask is then tranfered to the heating workstation [L], under ventilation. This operation is visualized by a video camera.

Ninth step :
After cooling, the teflon flask is transfered back to the workstation [H] to add fluorhydric acid (necessary to dissolve Si, W, Nb compounds).

Tenth step :
To adjust final concentration and acidity of the solution, water is added at workstation (G). After homogeneization (workstation [F]), the teflon flask is weighed (workstation [E]) to calculate the final concentration.

Eleventh step :
From the teflon flask, the robot fills two analytical flasks (at workstation [M]) and transfers them to the ICP rack (workstation [N]).
The left part of the solution is put to the waste in a special plastic bottle which is then evacuated using a lead cask.
The cleaning of the teflon flask is obtained by performing a complete cycle without any sample (acid mixture, heating, homogeneization...).

Twelfth step :
The ICP rack is manually transfered to the automatic sampler of the spectrometer and the analysis is performed.

Thirteenth step :
The container with the left part of the metallic sample is evacuated using a lead cask.

4.2. CHEMICAL AND RADIOCHEMICAL DECONTAMINATION

When the surface of the sample is dirty it's necessary to clean it before analysis. The following sequence is used to perform this operation :

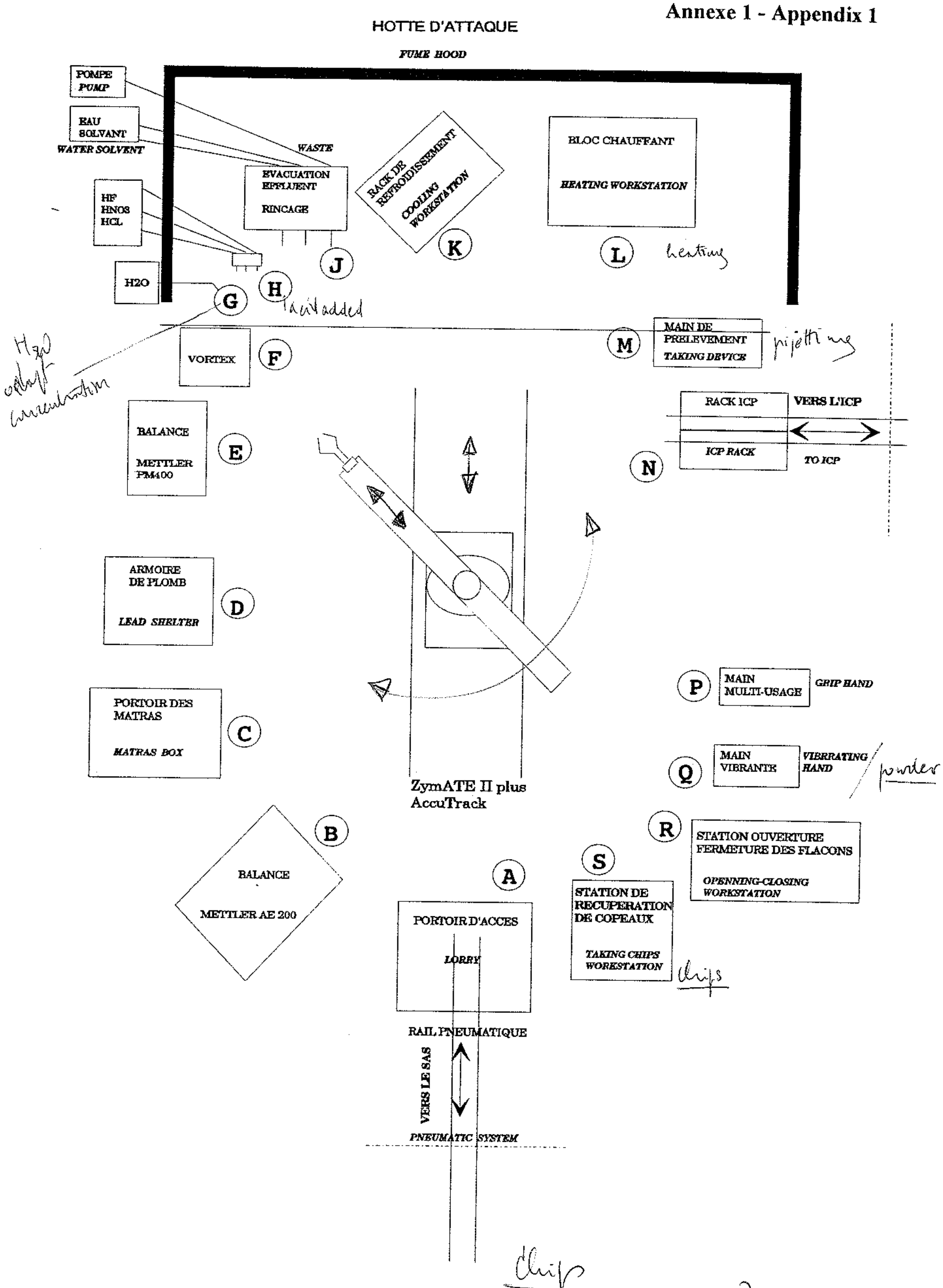
- introduction of the sample in the robot area,
- opening of the container,
- handling and weighing of the sample,
- adding of acid mixture (or cleaning product in the case of grease),
- heating,
- measurement of the dose rate and of the weight after treatment,
- sampling for analysis,
- the sample is ready for further treatment.

5 ZYMATE PROGRAMMING

Three ways of programming are available :

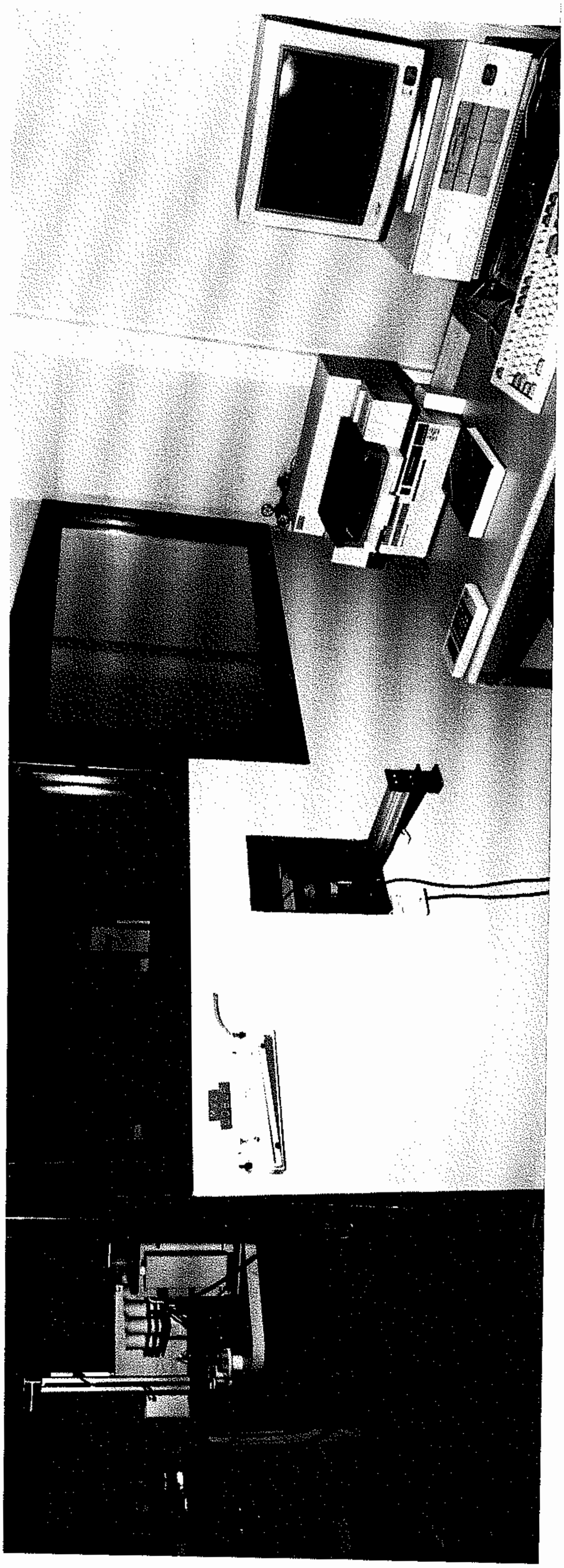
- macro-instructions programming,
- x, y, z, programming,
- the operator can execute a special movement using a remote command station and then memorize this motion on the computer.

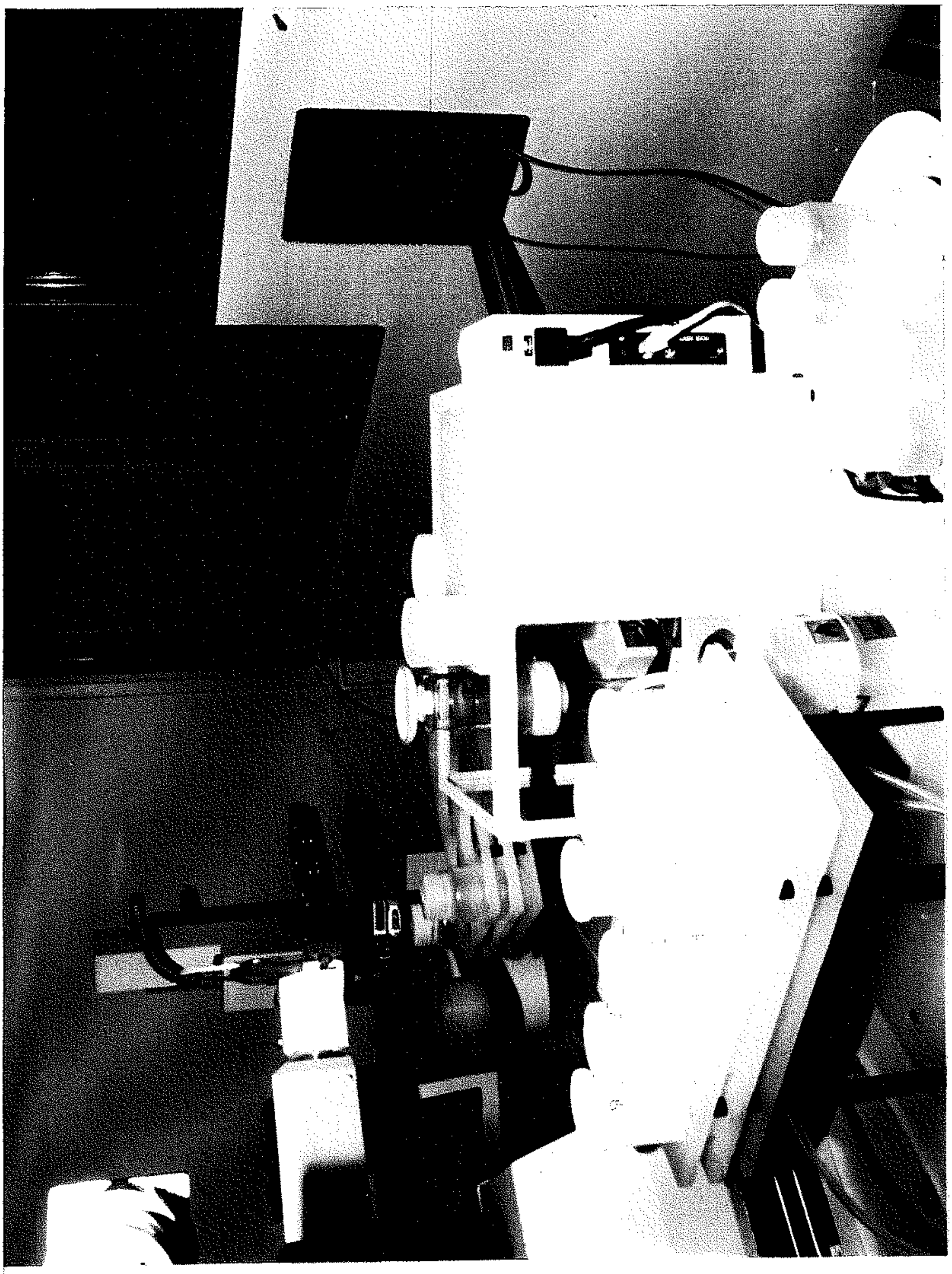
Annexe 1 - Appendix 1



chips
or fine powder in bottle

Annexe 2 - Appendix 2





Annexe 4 - Appendix 4