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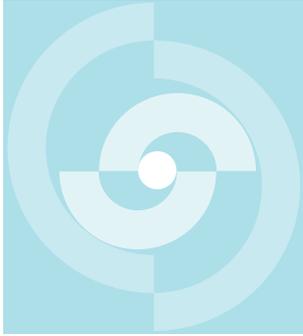
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# TECHNICAL REPORT 87-39

## 3-D MIGRATION EXPERIMENT-Report 2: Instrumentation and Tracers

Harald Abelin  
Lars Birgersson  
Jard Gidlund

November 1987

Department of Chemical Engineering,  
Royal Institute of Technology, Stockholm



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Der vorliegende Bericht betrifft eine Studie, die für das Stripa-Projekt ausgeführt wurde. Die Autoren haben ihre eigenen Ansichten und Schlussfolgerungen dargestellt. Diese müssen nicht unbedingt mit denjenigen des Auftraggebers übereinstimmen.

Le présent rapport a été préparé pour le projet de Stripa. Les opinions et conclusions présentées sont celles des auteurs et ne correspondent pas nécessairement à ceux du client.

This report concerns a study which was conducted for the Stripa Project. The conclusions and viewpoints presented in the report are those of the authors and do not necessarily coincide with those of the client.

Das Stripa-Projekt ist ein Projekt der Nuklearagentur der OECD. Unter internationaler Beteiligung werden von 1980-86 Forschungsarbeiten in einem unterirdischen Felslabor in Schweden durchgeführt. Diese sollen die Kenntnisse auf folgenden Gebieten erweitern:

- hydrogeologische und geochemische Messungen in Bohrlöchern
- Ausbreitung des Grundwassers und Transport von Radionukliden durch Klüfte im Gestein
- Verhalten von Materialien, welche zur Verfüllung und Versiegelung von Endlagern eingesetzt werden sollen
- Methoden zur zerstörungsfreien Ortung von Störzonen im Fels

Seitens der Schweiz beteiligt sich die Nagra an diesen Untersuchungen. Die technischen Berichte aus dem Stripa-Projekt erscheinen gleichzeitig in der NTB-Serie der Nagra.

The Stripa Project is organised as an autonomous project of the Nuclear Energy Agency of the OECD. In the period from 1980-86, an international cooperative programme of investigations is being carried out in an underground rock laboratory in Sweden. The aim of the work is to improve our knowledge in the following areas:

- hydrogeological and geochemical measurement methods in boreholes
- flow of groundwater and transport of radionuclides in fissured rock
- behaviour of backfilling and sealing materials in a real geological environment
- non-destructive methods for location of disturbed zones in the rock

Switzerland is represented in the Stripa Project by Nagra and the Stripa Project technical reports appear in the Nagra NTB series.

Le projet Stripa est un projet autonome de l'Agence de l'OCDE pour l'Energie Nucléaire. Il s'agit d'un programme de recherche avec participation internationale, qui sera réalisé entre 1980 et 1986 dans un laboratoire souterrain, en Suède. Le but de ces travaux est d'améliorer et d'étendre les connaissances dans les domaines suivants:

- mesures hydrogéologiques et géochimiques dans les puits de forage
- chimie des eaux souterraines à grande profondeur
- écoulement des eaux souterraines et transport des radionucléides dans les roches fracturées
- comportement des matériaux de colmatage et de scellement des dépôts finals
- méthodes de localisation non destructive des zones de perturbation de la roche

La Suisse est représentée dans le projet Stripa par la Cédra. Les rapports techniques du projet Stripa sont publiés dans la série des rapports techniques de la Cédra (NTB).

**ABSTRACT**

This report is one of the four reports describing the Stripa 3D experiment where water and tracer flow has been monitored in a specially excavated drift in the Stripa mine. The experiment was performed in a specially excavated drift at the 360 m level in granite. The whole ceiling and upper part of the walls were covered with more than 350 individual plastic sheets where the water flow into the drift could be collected. 11 different tracers were injected at distances between 11 and 50 m from the ceiling of the drift. The flowrate and tracer monitoring was kept up for more than two years. The tracer breakthrough curves and flowrate distributions were used to study the flow paths, velocities, hydraulic conductivities, dispersivities and channeling effects in the rock.

The present report describes the instrumentation developed and used as well as the tracers that were tested and used in the experiment.

RESUME

Ce rapport est l'un des quatre rapports décrivant l'essai 3D de Stripa où les circulations d'eau et de traceurs ont été enregistrées dans une galerie spécialement excavée dans le granite au niveau 360 m dans la mine. Tout le plafond et la partie supérieure des parois de la galerie ont été recouverts par plus de 350 feuilles individuelles en plastique permettant de recueillir les entrées d'eaux dans la galerie. 11 traceurs différents ont été injectés à des distances se situant entre 11 et 50 m du plafond de la galerie. Les mesures de débit et la détection des traceurs ont été maintenues durant plus de deux années. L'irruption des traceurs et la distribution des débits ont permis d'étudier les cheminements d'eau, les vitesses d'écoulement, les conductivités hydrauliques, les dispersivités et les effets de "channeling" dans la roche.

Le présent rapport décrit l'instrumentation mise au point et utilisée ainsi que les traceurs qui ont été expérimentés et utilisés durant l'essai.

### ZUSAMMENFASSUNG

Dieser "Report" ist einer von gesamthaft vier Berichten, die das 3D-Experiment in der Stripa-Mine beschreiben. In einem neu erstellten Stollen auf der 360 m-Sohle wurden Wasser- und Tracerfluss im Granitgestein beobachtet. Die ganze Stollendecke und der obere Teil der Stollenwände wurden mit über 350 einzelnen Abdeckfolien ausgerüstet, um den gesamten Wassereintrag in den Stollen zu ermitteln. 11 verschiedene Tracers wurden in Abständen von 11 bis 50 m von der Stollendecke ins Gestein injiziert. Die Fliesszeiten des markierten Wassers wurden über einen Zeitraum von mehr als zwei Jahren registriert. Die Tracer-Durchbruchskurven und die Fliessratenverteilungen ergaben Daten über Fliesswege, Wassergeschwindigkeiten, hydraulische Leitfähigkeiten, Dispersivitäten und Kanaleffekte im Gestein.

Dieser Bericht beschreibt die entwickelte Instrumentierung sowie die während des Versuchs getesteten und verwendeten Tracers.

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## SUMMARY

Equipment for a large scale tracer test has been developed. Different conservative tracers have been injected with a constant over pressure into conductive zones found in three vertical bore holes. The length of each injection zone was 2.5 m , the rest of the holes were sealed with a packer system which had bentonite as the sealing component. The advantage of the bentonite packer system is its independence on external supplies i.e. it will not fail with time but will instead increase its sealing capacity. The major disadvantage is the short time available for installation of the packer system before it starts to swell.

Water sampling was performed by completely covering the upper part of the drift from which the three vertical injection holes were drilled, with 350 plastic sheets each 2 m<sup>2</sup>. With this sampling arrangement all water entering the upper part of the test site was sampled for tracers and monitored to give the water flowrates.

The water from each plastic sheet was sampled during preset time intervals by fractional collectors. The rest of the time it was led to flasks so that the individual flowrate to each sheet could be determined. To be able to monitor the water inflow to the uncovered parts of the test site, two walls were constructed, one at the entrance of the test site and the other one at the entrance of the access drift leading to the test site. At these walls air velocities, temperatures and humidity were monitored to give the amount of water added to the ventilation air coming out from the test site.

The 9 used conservative tracers i.e. nonsorbing, were selected from nearly 100 tested dyes and salts. After a first coarse test the remaining 40 were tested for long time stability and sorption on granite and materials used in the equipment. Those of the injected tracers that were found at the sampling points in the test site were also tested in through diffusion experiments to give information on the effects of matrix diffusion for each of the tracers.

## ACKNOWLEDGEMENTS

This work was performed within the OECD/NEA International Stripa Project managed by the Swedish Nuclear Fuel and Waste Management Co. We gratefully acknowledge the financial support and the never failing encouragement and help from the initiator of the project Lars B. Nilsson and the project manager Hans Carlsson. Great thanks are due to the chairmen of the technical subgroups, Rudi Beck and Paul Gnirk, and the members of TSG1 for their support and constructive advice.

We would also like to thank Clarissa Villalobos for performing the analysis in an accurate and well documented manner and Margareta Lundberg for preparing the reports.

## INTRODUCTION

This report is one of the four reports and three stand alone appendices which make up the final report of the Stripa 3-D project. The series consists of the following reports:

1. *Site preparation and documentation*
2. *Instrumentation and tracers*
3. *Performed experiments, results and evaluation*
4. *Fracture network modelling of the Stripa 3-D site.*

"Stand alone" appendices:

- *Tracer breakthrough curves*
- *Fitted tracer curves*
- *Flowrate curves and tracer breakthrough curves for individual sheets - smoothed data.*

### **BACKGROUND**

In many countries the final repositories for high level radioactive waste are planned to be located at large depths in crystalline rock formations. The only known means for the radionuclides to migrate to the accessible environment is by flow with mobile water in the fractures in the rock and by molecular diffusion. The latter process can be neglected as an escape mechanism for the depths considered. It is known that the rock is fractured and that practically all water movement is confined to the fractures. The rock matrix is porous and may be accessed by diffusing species but due to the low hydraulic conductivity of the matrix, flow is negligible under repository conditions.

To assess the potential release and transport of the radionuclides, information on the flowrates, velocities and on the pathways of the water is needed. The nuclides which are dissolved in water will be able to diffuse into the micropores of the porous rock matrix and so be withdrawn from the flowing water in the fractures. Sorbing nuclides will sorb on the surfaces of the fractures and also on the inner surfaces of the rock matrix which are accessed by diffusion. The rate of uptake into the rock matrix will thus be directly influenced by the size of the fracture surfaces that are wetted by the mobile water.

Only a small fraction of the fractures in the rock is open to flow. In the Stripa 2-D experiments it was also observed that the water flow was unevenly distributed in the fractures (Abelin et al., 1985). Such channeling effects may give rise to preferential fast pathways and limit the wetted area.

The velocity variations between fractures or channels may cause dispersion of the migrating radionuclides both in the longitudinal direction and in the transverse direction. Longitudinal dispersion may cause dilution but may also allow some part of the nuclides to arrive earlier than the main portion and thus allow less time for decay. Transverse dispersion may also give dilution.

## 1.2 SOME FEATURES OF THE THE PRESENT INVESTIGATION

Very few investigations have been made regarding flow and transport properties in deep lying crystalline rocks. There are no well developed techniques available to investigate the properties of interest. The flow patterns and mechanisms are not well known and the investigative techniques must be developed so that many conceivable results can be accommodated with the experiments.

The flowrates, velocities, channeling frequencies and geometry of pathways were not know before the experiment to within several orders of magnitude. Nor were the possible variations in the properties known or could be reasonably assessed.

To accommodate possible large variations of these properties some special techniques were developed. The basis for the whole experiment was the development of the plastic sheeting technique which permitted a very detailed monitoring of the water flowrate and tracer path distribution. Another special aspect of the experiment was to use as many different tracers as possible, 9 tracers, in order to investigate variations in the transport properties of the rock. The choice of different injection distances was made with dual purposes. First, because of the a priori unknown water velocity a large difference in the migration distance would ensure that at least some of the tracers would arrive during the duration of the experiment. Second, different migration distances along essentially the same flow path may give information on how the dispersivity is influenced by the migration distance. This is an open question of some importance.

The size of the wetted surfaces is unknown. If it is large, small molecular weight tracers might diffuse into the micropores of the matrix and to a large extent be withdrawn from the mobile water. This might cause the tracers to be retarded and diluted to such an extent that detection in the collected water in the drift may be difficult. Tracers of different molecular weight were used and a high molecular weight tracer was synthesized especially for this purpose.

## 1.3 AIMS

The broader aims of the investigation have been discussed above. The detailed aims are summarized below:

- \* Develop techniques for large scale tracer experiments in low permeability fractured rock
- \* Determine flow porosity
- \* Obtain information on longitudinal and transverse dispersion

- \* Obtain information on channeling
- \* Obtain data and other information for validation and/or modification of models.

2

## DESIGN OF EXPERIMENT

2.1

### REQUIREMENTS

The following basic requirements were used for designing the actual experiment:

1. The experiment should be carried out in "undisturbed rock"
2. It should be possible to monitor the longitudinal as well as the transverse spreading of the injected tracers
3. It should be possible to continuously inject conservative tracers into some conductive fractures within the rock block of interest over a long period of time
4. The injection flowrates should be small compared with the natural flowrates
5. Only conservative tracers should be used.

The term "undisturbed rock" in Requirement 1 does not mean the disturbance induced by the mine but rather disturbances, such as drifts and/or boreholes in the vicinity of the rock block, which could act as local hydraulic sinks. It was decided that there should not be any hydraulic sinks, other than the test site, closer to any injection point than twice the distance from injection point to collection point, which was planned to be at most about 50 m. Due to the requirement of the undisturbed rock it was decided that the amount of drilling within the rock block of interest should be kept at a minimum, i.e. only the injection holes were drilled prior to the tracer test. If it was found necessary to drill any investigation boreholes, e.g. to determine "boundary conditions", then these were to be drilled after the experiment.

2.2

### LAYOUT AND LOCATION

To make it possible to monitor the transverse dispersion of the tracers, as mentioned in Requirement 2, the sampling had to be done over a large area and not only at a few spots. Experience from the "Migration in a Single Fracture" experiment (Abelin et al., 1985) led to the decision to excavate a drift within the rock block of interest to enable sampling over a larger area. This drift would also act as a hydraulic sink.

To fulfill Requirement 1, a 115 m long access drift had to be excavated before the actual excavation of the test site could be performed. A map of the test site location within the mine is given in Appendix 1. The excavation of the test site was done by soft blasting.

Before the actual excavation of the drift, a pilot hole was drilled to make sure that there were no major unacceptable features within the

area where the test site was to be located. This pilot hole is marked 3D-P1 in Figure 2-1.

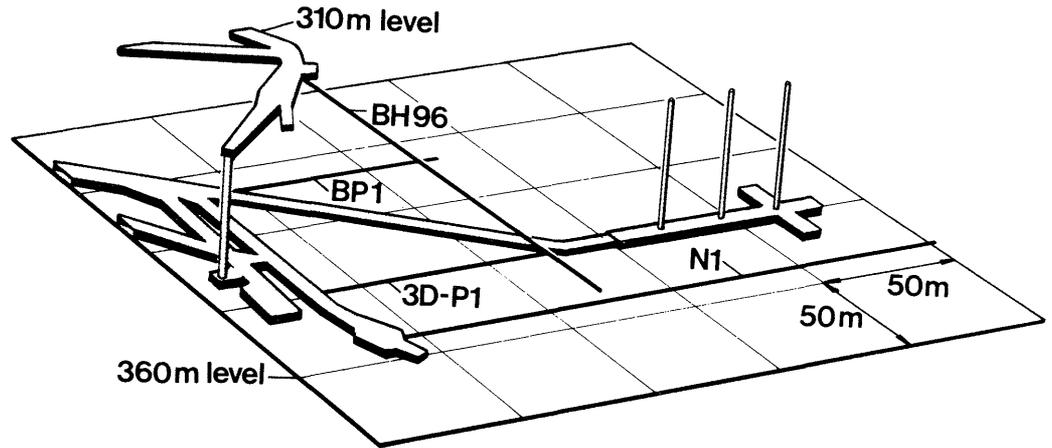


Figure 2-1. Map over test site and adjacent areas.

The layout of the drift is shown in Figure 2-2. The drift is in the form of a cross to enable sampling in two directions. The actual water collection was done by covering the upper part of the drift, approximately 700 m<sup>2</sup>, with plastic sheets, each with an area of 2 m<sup>2</sup>, giving a total of 350 collection points.

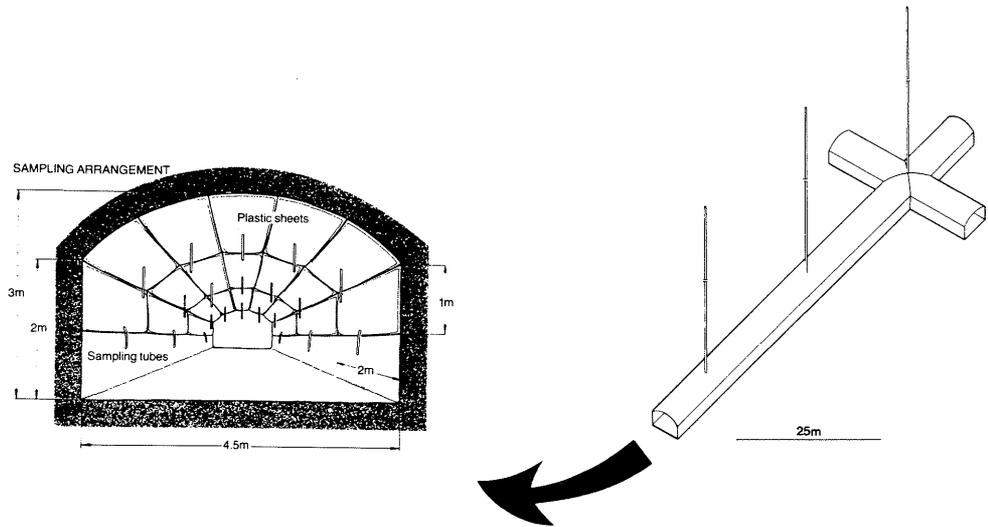


Figure 2-2. Layout of the test site with injection holes.

From the test site, three vertical holes were drilled for the injection of the tracers. The decision where to place the injection zones was based on the results from the outflow measurements performed in 2 m intervals. The injection of the nine different tracers was done at nine different zones, each zone 2.5 m long, at a maximum distance of about 50 m from the drift. The tracers were injected over a time period of 2 years. Even though the maximum distance was to be 50 meters, it was decided that the injection holes should be 70 m long. The extra 20 meters were used to obtain information on the rock closest to the furthest injection points. The parts of the injection holes not used for injection were sealed off. Different conservative tracers were used in the nine injection zones. The time interval of the water sampling was adapted to the flowrate at the sampling point. At most sampling points within the test site water samples were taken every 16 hour.

### 3 EQUIPMENT

#### 3.1 GENERAL

The design of the equipment both in the laboratory and in the field is based on the experience gained from the "Migration in a Single Fracture" (2-D) experiment which was a part of the STRIPA PROJECT Phase I. Most of the laboratory equipment used in the 2-D experiment was used in this experiment but with slight modifications. The field equipment had to be redesigned due to the difference between the two experiments.

The field equipment is divided into five major parts:

- Packer system with injection zones
- Equipment for injection outside the injection hole
- Equipment for water collection
- Pressure monitoring system
- Equipment used in the "ventilation test."

This five groups are treated under separate headings below.

#### 3.2 REQUIREMENTS

The overall requirement for the field equipment was that it should be reliable over a long period of time and be almost independent of the main voltage supply. The analyzing equipment used in the laboratory should be able to handle 1000 water samples a week.

The materials used in the equipment should not release compounds that would interfere with the analysis or interact with the tracers. However, the plastics that were used, PVC and Nylon, released small amounts of softener which completely blocked the light absorbance spectrum below 300 nm.

#### 3.3 FIELD EQUIPMENT

##### 3.3.1 Packer system

The packer system was to seal off the complete length of the borehole except for the 2.5 m long injection zones. Having 70 m long injection holes and an average of three injection zones in one injection hole, the packer system had to seal off 63.5 m of the borehole. Due to the geometry of the drift and the packer pusher system, no part of the packer could be longer than 1 m. To have 63 inflatable packers within a single hole and a total of 190 packers inflated over at least a year seemed to be too great a risk and to replace the packers with mechanical packers would be too costly. Therefore new methods to seal

the boreholes had to be considered. One suggestion by Pusch (1983) was to use bentonite in sealing off the injection holes.

Bentonite is a clay which swells when in contact with water, so when used in a packer system it will swell and seal off areas in the holes where water enters. Areas where no water enters need not be sealed off and will not be sealed off. One other advantage of the bentonite is that the sealing effect improves with time. The disadvantages are that the packer system is not removable and there is a limited time for installation before the bentonite starts to take up water and expand.

Figure 3-1 shows a schematic drawing of the bentonite packer system designed for this experiment. The system consists of four major parts: (1) supporting rod system which contains tubing, (2) compacted bentonite, (3) injection zones, and (4) extra mechanical sealing to prevent the bentonite from reaching the injection zone.

To reduce the risk of the bentonite appearing in the injection zone and thereby being injected into the fracture of interest, the injection zone was straddled in series with a swelling cloth and mechanical packers. The first contamination of bentonite occurs before the actual swelling of the bentonite because the injection hole is completely filled with water which contains small amounts of bentonite particles. To reduce this early contamination of bentonite, the swelling cloth was used, see Figure 3-1. The cloth expanded immediately when it comes in contact with water. However, the cloth was not able to sustain the pressure of the swelling bentonite. To stop the later contaminant due to the swelling bentonite the mechanical packer was used which was compressed by the swelling bentonite.

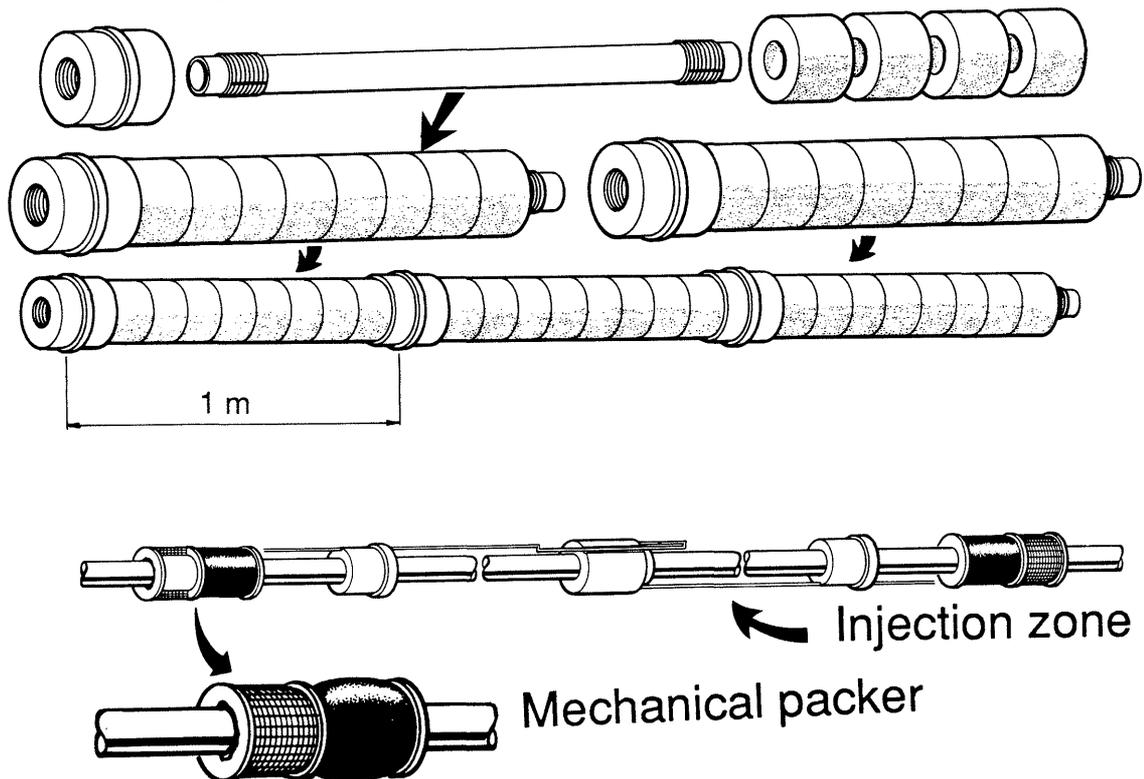


Figure 3-1. Schematic drawing of bentonite packer system.

A prototype of the bentonite packer was built and tested in the laboratory. The laboratory setup is shown in Figure 3-2. In these tests it was possible to maintain a water pressure difference of 15 bars over a distance of 25 cm of bentonite. The effect of the sealing mechanical packers on both sides of the injection zone could not be studied due to the difficulties of applying additional water when the swelling of the bentonite had started.

From practical field tests performed in injection holes I and II, it was estimated that the available time for installing the packer system would be approximately 3 hours.

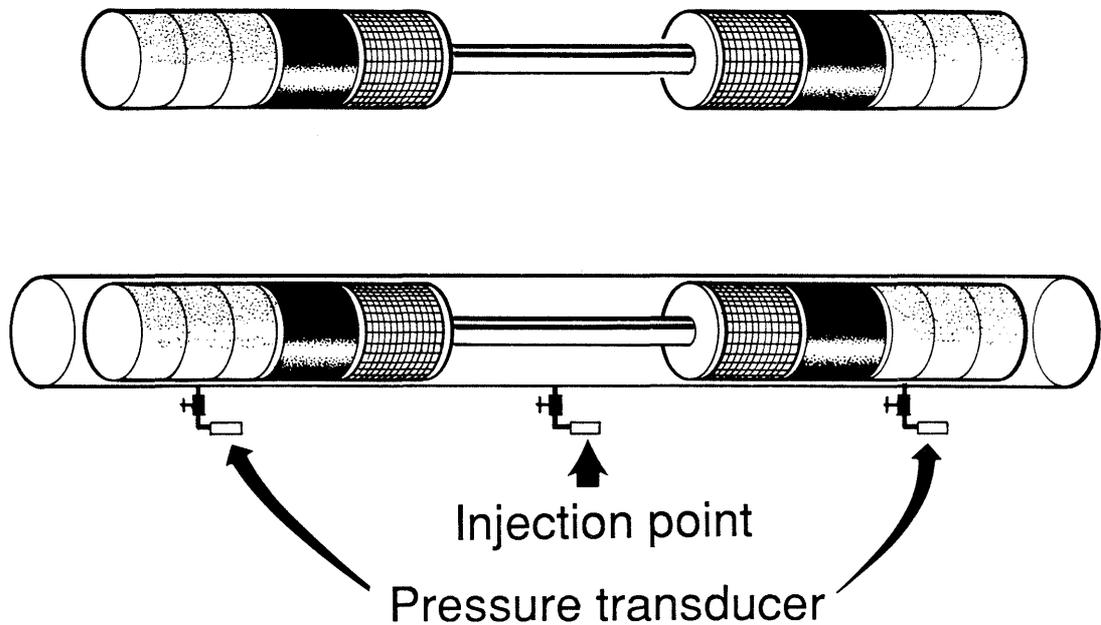


Figure 3-2. Laboratory setup to test bentonite packer.

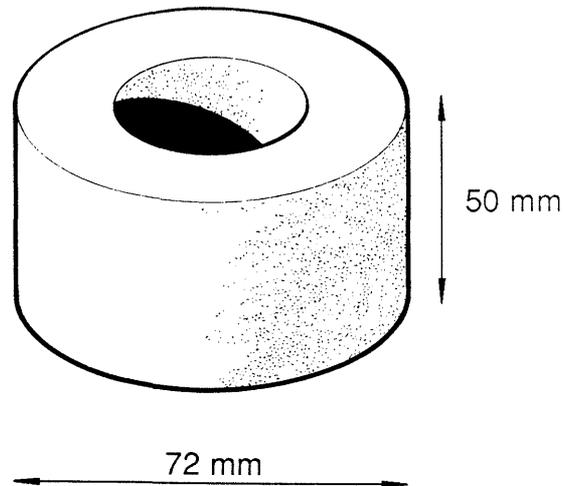


Figure 3-3. Compacted bentonite used in the packer system.

The swelling pressure of the bentonite is dependent on the ratio of free volume to volume of bentonite and on the density of the compacted bentonite. The dimensions of the bentonite elements are given in Figure 3-3.

The bentonite was compacted to a density of  $2.2 \text{ kg/dm}^3$ . Due to the large diameter of the supporting rod of the packer system, which contained up to 9 nylon tubes, it was not possible to have any outer mechanical protection for the bentonite without obtaining a bad ratio of the free volume to volume of bentonite. It was instead decided that at every meter there would be stainless steel supports with a diameter slightly larger than that of the bentonite. These stainless steel supports eliminated all contact between the bentonite and the walls in the borehole during the installation of the packer system.

Each injection zone had an inlet at the bottom and an outlet at the top. At the top of the packer system there was also a single inlet. To be able to install the packer system, no part of the packer could be longer than 1 meter due to the limitations of the packer pusher system. The interconnections of the 1 m parts were sealed off by double O-rings within the stainless steel support, see Figure 3-4. Figure 3-5 shows the device used at the lower point in the injection zone which allowed the tubes to enter the injection compartment.

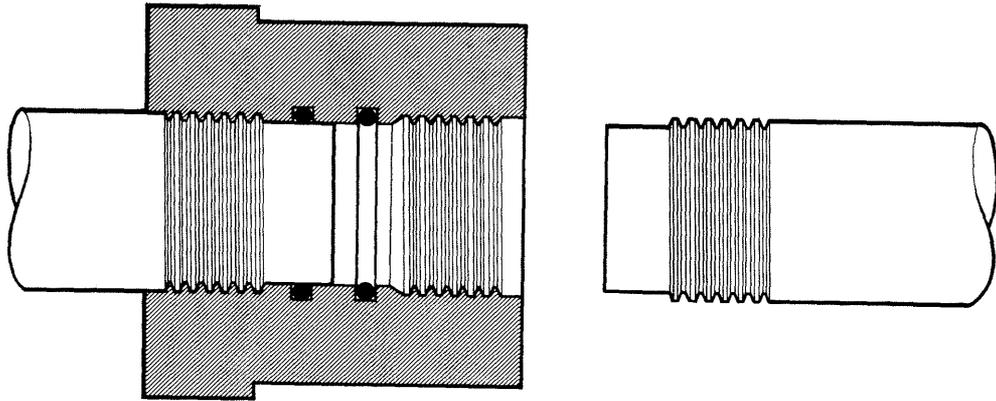


Figure 3-4. Stainless steel support used to join supporting rods.

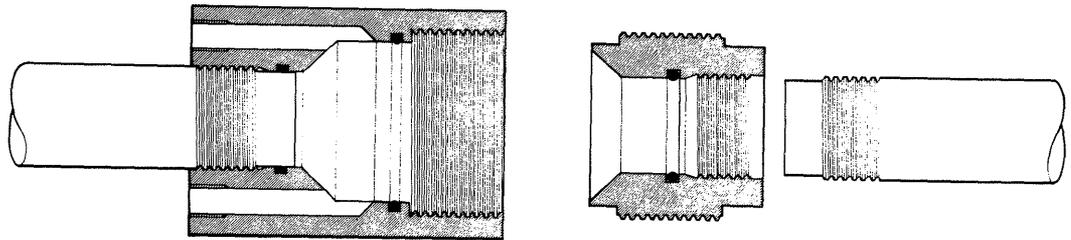


Figure 3-5. Lower section of one of the injection zones of packer system.

At the outer end of the packer system, an inflatable packer was used to seal off the hole at the face of the drift. This sealing at the outer end was used to keep the vertical hole filled with water until the bentonite had swelled.

When installing the bentonite packer system, a THORAM drilling equipment was used to push the packer system up into the vertical holes. Before installing the system, the complete packer for one hole was prepared, see Figure 3-6.

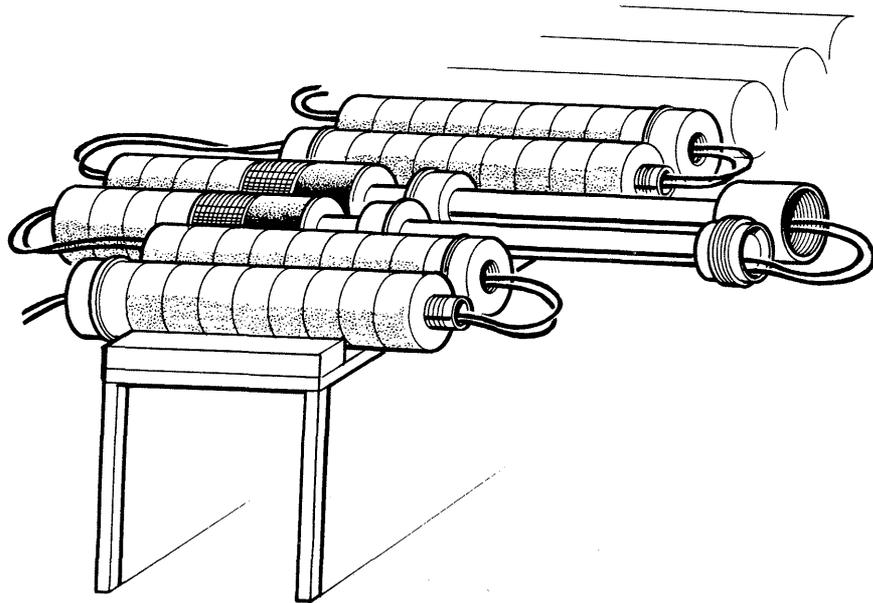


Figure 3-6. Preparation of packer before installation.

To assemble the packer system, all its elements were placed out together. The distance elements were positioned so that the injection zones were at the correct places and then the system was fitted with bentonite. After all the parts were laid out and fitted, tubing was pulled through the system and connected at the injection zones. The packer system resembled "a large chopped up snake" with only the tubes connecting the individual parts. The 1 m parts were put in the "packer pusher" and pushed into the holes just leaving the pipe thread at the end outside the hole. The next part was pulled over the tubing and connected and then the system was pushed further into the hole. This was repeated until the complete packer was installed. The "packer pusher" held the packer system in place until the bentonite had swelled enough to support the weight of the system. The system was then bolted to the rock at the face of the drift.

In both injection holes I and II it was possible to install the complete packer system without any interruption. In injection hole III it was suspected that the high water inflow rates would cause an early swelling of the bentonite which would endanger the installation. It was decided that the packer system should be installed in two parts, one inner part (20 m long) and a second part which contained all the injection zones. The first part was put in place and left for the bentonite to swell. After this was completed the second part was installed. This split up of the packer system into two parts had two

effects: (1) the first part sealed off the inner most part of the injection hole and thereby reduced the amount of water which would have come in contacted with the second part of the system during installation, and (2) the second part of the system was only 50 m long which reduced the time needed for installation.

### 3.3.2 Equipment for injection

Two methods of injection was considered: (1) injection with a constant flowrate, which would cause an increase in the injection pressure with time; and (2) injection with a constant pressure which would give a decrease in the injection flowrate with time. The constant injection flowrate would be maintained by pumps and in the case of constant injection pressure it would be supplied by compressed nitrogen.

In earlier experiments, Abelin et al., (1985) both used the injection methods with constant pressure and constant flow. The results from these different methods of injection showed that at low flowrates, down to 1 ml/h, using commercially available pumps were not reliable. In the case of injection with a constant pressure, the problem was to keep the variation of the injection pressure within acceptable levels. It was observed that injecting with a pressure close to the natural pressure, small variations in the injection pressure caused large variations in the injection flow. In addition, changes in the ambient temperature of a few degrees affected the pressure regulators enough to cause flow variations of  $\pm 50\%$ . On the other hand, the injection method with constant pressure was independent of the main power supply, so during a power failure the injections could continue. To reduce the variation in injection pressure mentioned above, two regulators were used to regulate the injection pressure. One crude regulator was positioned close to the nitrogen supply followed by a second finer regulator. The nitrogen flow was set by two precision needle valves in series. The system used to apply the constant injection pressure is shown in Figure 3-7.

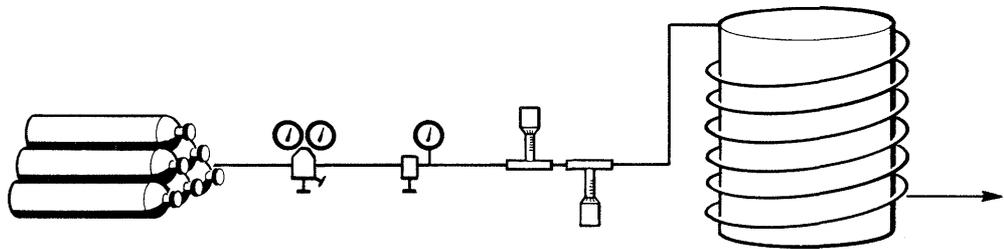


Figure 3-7. System used to apply constant injection pressure.

Figure 3-8 shows a schematic picture of the complete injection system which allowed for sampling of the tracer solutions at the different injection points. The injection system allowed for the circulation of the volume within the injection zone. The injection flowrates were monitored in the same way as in the earlier experiment by Abelin et al., (1985). In their experiment a 500 m long nylon tube, total volume 5 l, was wrapped around a paper barrel, see Figure 3-8. Nitrogen was supplied at the top during the injection thus forcing the tracer solution down in the tube. The distance traveled by the intersection of the nitrogen with tracer solution between the two readings gave the injected volume and the time that elapsed between the readings gave the flowrate. During the refilling of the tube, the top was connected to atmospheric pressure and the tracer solution was pumped into the bottom. Stock solutions of the different injection solutions were kept in polyethylene containers with a volume of 600 l. Prior to the start of injection, water was collected from the injection holes. This water was mixed with the tracers and reinjected. Thus the injected water was water which had been obtained from the injection holes.

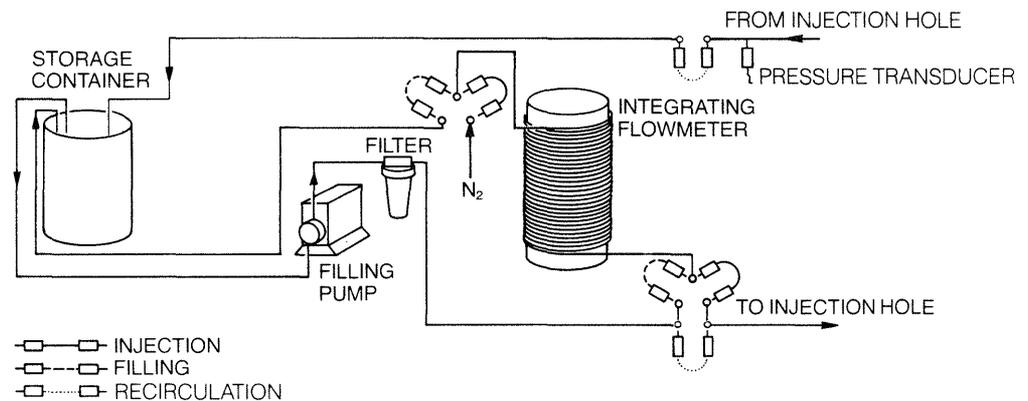


Figure 3-8. The injection system.

Each injection zone was equipped with two barrels, one was used for injection while the other one could be refilled. At one of the injection points in injection hole III there was a leakage in the injection zone into the supporting rod of the packer system. The injected water that emerged at the end of the packer system at the face of the drift was collected and the flowrate monitored to make it possible to determine the actual injected volume. Due to this leakage there was a high "injection" flowrate and the ordinary injection system could not be used. Instead water was injected by a membrane pump equipped with a pressure sensing device which turned the pump on and off. To

reduce the pulsating in the pressure when the membrane pump was working, a barrel with a 500 m long tube was connected between the pump and the injection zone.

### 3.3.3 Equipment for water collection

The water entering the upper part of the test site was collected in plastic sheets glued to the rock surface. Connected to each sheet was a PVC tubing through which the water was collected. The advantages of this method compared with drilling shallow sampling holes to collect the water were: (1) the method was cost effective, many collection points were obtained at a low cost; and (2) the risk of missing some of the water entering the sampling area was eliminated. A drawback of the method is that changes in the rock stress due to the excavation of the drift could change the flow pattern close to the drift, therefore locally obtained flow patterns may not represent the flow in the undisturbed rock.

The actual configuration of the plastic sheets is given in Figure 3-9. Each of the plastic sheets had an area of about  $2 \text{ m}^2$ . The technique of gluing the sheets set the maximum sheet size to  $2 \text{ m}^2$ , larger sheet sizes could not be sustained by the glue before hardening. Different glues and plastic materials were tested for suitability and it was found that plastic films made out of PVC, 0.1 mm thick, was preferred to films made of polyethylene. The PVC material is somewhat more stretchable and could thereby be easily fitted to the rough walls of the drift. It also works well with the glue, GOODRICH PL 400 which was used in gluing the plastic sheets.

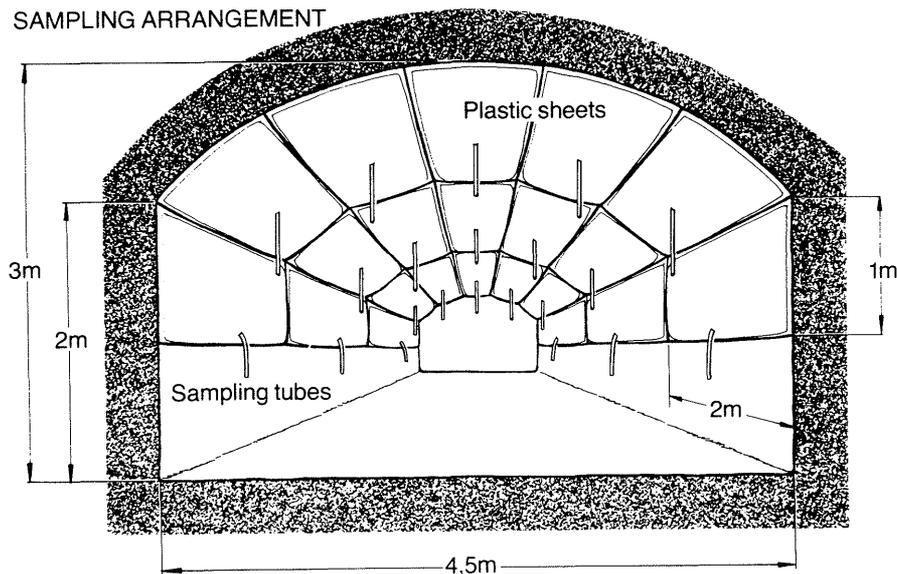


Figure 3-9. Cross section of test site with water sampling arrangement.

To facilitate the installation of the sheets, the drift was measured using a laser device and squares painted on the face of the drift. The gluing was not done directly on the paint, but a few centimeters

beside, so not to risk any synergistic effects between the glue and paint which would reduce the long time stability of the joint.

The two major problems with gluing the plastic sheets to the walls of the drift were:

- The walls were covered by a thin dust layer
- Parts of the rock surface were moist.

These problems were overcome by washing the walls with a high pressure cleaner and then, before applying the glue, drying the wet areas with a gas burner. The plastic sheets should overlap in such a way that eventual leaking water enters the sheet below. To help support the plastic sheets before the mounting glue had hardened, melting glue was used at a few spots.

To determine the number of sampling points, the plastic sheets were left these undisturbed for a few weeks to allow these accumulate water. From this procedure, it was determined which sheets needed to be sampled and also where to apply the drainage apparatus. The drainage apparatus was a long plastic tube glued to the plastic sheet by melting glue. The actual steps for mounting the drainage tube to the plastic sheets are shown in Figure 3-10.

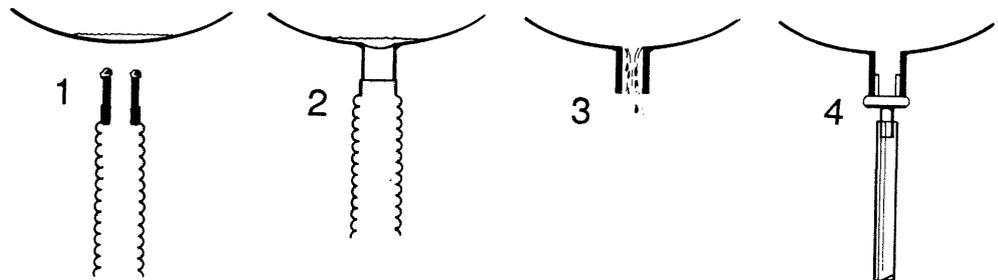


Figure 3-10. Mounting of drainage tube to plastic sheets.

One problem with the plastic sheets was that the sheets made up a large area through which water could be transported away by diffusion. Tests in the laboratory show that the mass transport rate of water is about 1 ml/h;m<sup>2</sup>. The relative humidity was measured in the laboratory to be 40 - 50 % at an ambient temperature of 20°C which should be compared with a relative humidity of 70 - 80 % at 15°C in the test site, i.e. the driving force is larger in the laboratory experiment in comparison with the real situation. Proportioning the measured value in the drift with both the driving force for diffusion (relative humidity difference) and the vapor pressure, a diffusion rate of 0.3-0.4 ml/h;m<sup>2</sup> was obtained. This applies only to the wet sheets. In the dry sheets the driving force was unknown. If all the sheets are saturated, a maximum diffusional loss 210-280 ml/h is possible. However, if only those sheets which are visibly wet are accounted for, a minimum loss of 70-100 ml/h is expected.

The tubes extending from the plastic sheets were connected to fractional collectors. To reduce costs, tubes from several sheets were connected to the same fractional collector. But this caused a problem in selecting a suitable time interval for sampling due to the different flowrates. To solve this problem, individual flow switches were installed on each tube. Each flow switch had its own time-relay so that the filling time of each test tube could be set. The switches were designed to give no pressure drop and no internal hold up. A schematic drawing of the flow switch is given in Figure 3-11.

The racks for the test tubes were the same as those used in the automated analyzing equipment at the laboratory. During the transport from the test site to the laboratory, the racks containing the water samples were kept in special containers made from paper cylinders. These cylinders reduced the effect of varying conditions during the transport of the samples. Figure 3-12 shows a set of racks and the container used in transporting the samples.

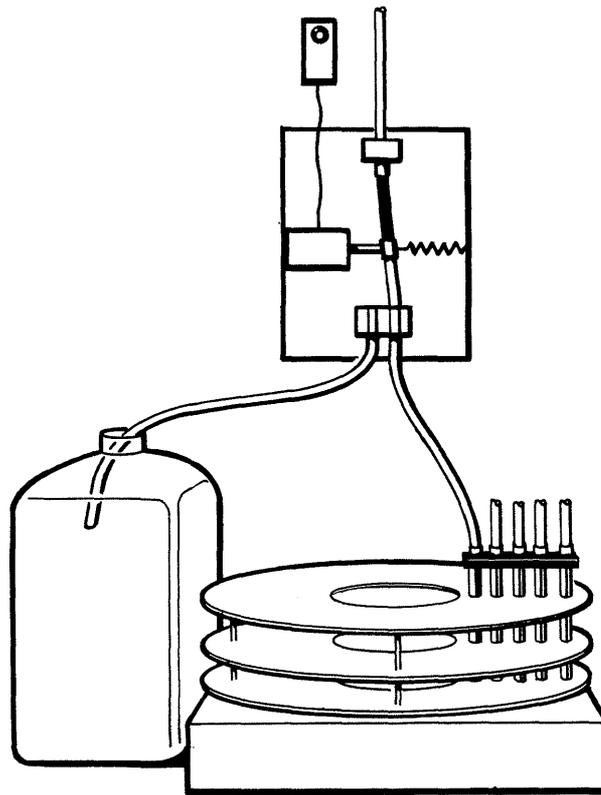


Figure 3-11. Flow switch used in the water collecting system.

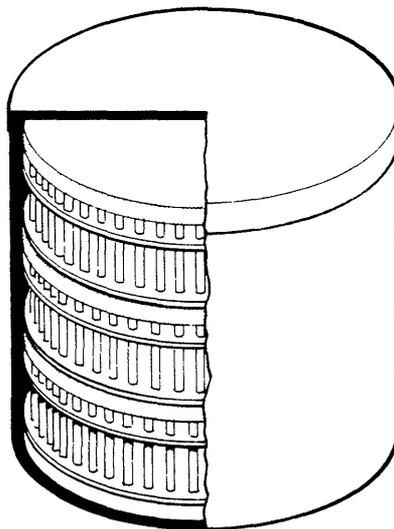


Figure 3-12. Test tube racks with transport container.

### 3.3.4 Pressure monitoring system

The pressure monitoring system was used to monitor the pressures at 12 different points, the 9 different injection zones and at the top of each of the three injection holes. The system consisted of pressure transducers with built in amplifiers. These amplifiers made it possible to have long connections (up to 70 m) between the transducer and the data acquisition system. The transducers were supplied with a stable voltage from a HP6205C (HP stands for Hewlett Packard). The data acquisition system consisted of a HP85 computer to which a HP3497A scanner was connected. Pressures were sampled every 2 hour.

To spare the membranes in the pressure gauges, a protecting layer of silicon oil was applied between the water and the membrane. The mounting of the pressure transducers is shown in Figure 3-13. The arrangement of the pressure monitoring system allowed for an easy calibration of the pressure transducers without having to remove them from the system.

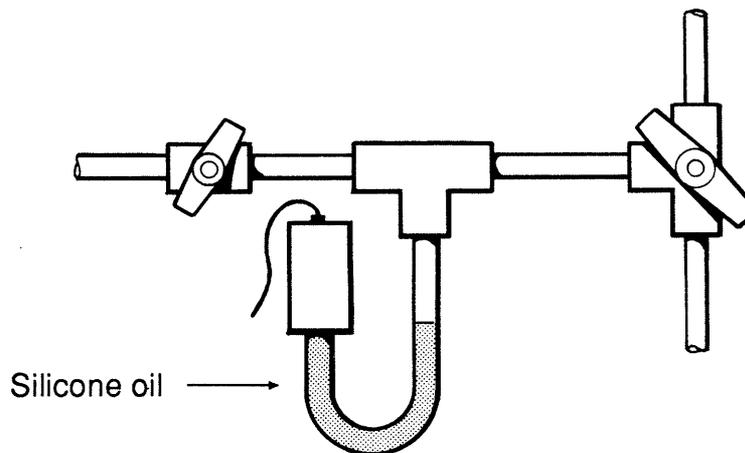


Figure 3-13. Mounting of pressure transducers.

### 3.3.5 Equipment and setup used in the "ventilation" experiment

After the injections had started, it was later decided that the total inflow to the test site and not only the inflow to the upper part of the drift should be monitored. It was predicted that the total inflow to the uncovered part of the drift could be from one liter per hour up to a few liters per hour. This flow range determined which method and equipment to use in monitoring the water flowrates.

The inflow to the uncovered areas was monitored by measuring the amount of water transported by the ventilation air in and out from the test site. These measurements were performed at two locations: (1) at the wall located at the entrance of the test site, and (2) at the wall constructed for the ventilation experiment at the beginning of the access drift. The wall locations are shown in Figure 3-14. At both walls 4 m long ducts with diameters of 50 cm were installed to allow for stable measuring conditions. The average velocity was calculated using the local velocity at five different spots within the same plane in the duct.

At first velocity, humidity, and temperature were monitored both at the inlet duct and outlet duct. Comparing the air flowrate results from the inlet and outlet showed that velocity monitoring needed to be measured only at the outlet duct. At the same time as the velocity was measured, 4 individual readings of the air temperature and humidity were performed.

The air velocity was measured by a TESTOVENT 4300 which has a measuring span of 0-40 m/s with a resolution of 0.01 m/s and an accuracy of  $\pm 1$  digit.

Air temperature and humidity were both measured with a HYGROTEST-6200 with a 6200-6250 probe. The resolution of temperature was 0.1 °C and the accuracy  $\pm 0.3$  °C. The accuracy for the humidity measurements was  $\pm 2\%$  of the relative humidity over the hole range.

The measured air velocities were approximately 10 m/s with differences in the humidity between the inlet and outlet of a few percent up to 5 %. Temperature differences range from 1 °C up to 4 °C. At the outer wall, the temperature differences were small but the differences in the relative humidity were larger. The opposite was observed at the inner wall, there were large differences in temperature and smaller differences in the humidity.

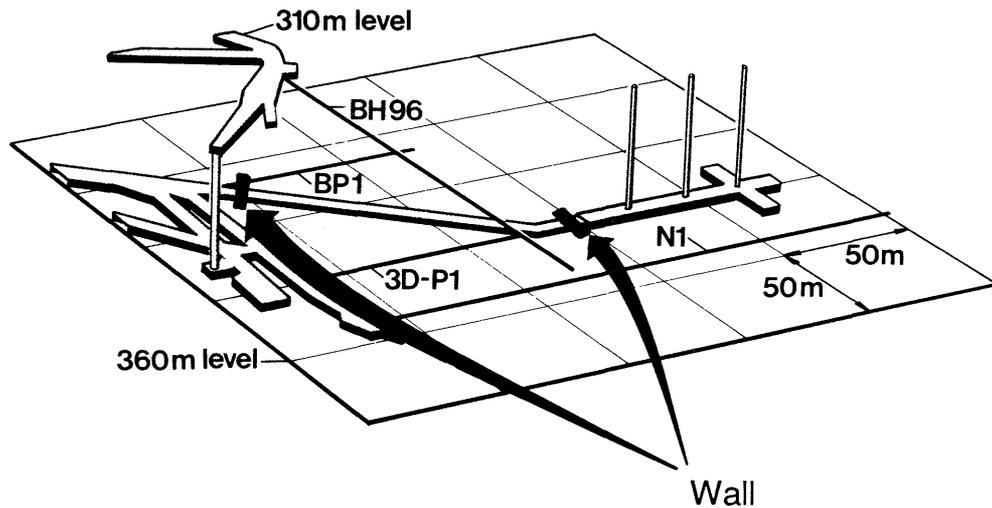


Figure 3-14. Location of monitoring points for total water inflow.

### 3.4

#### Laboratory equipment

##### General

The water samples were taken to the laboratory at the Royal Institute of Technology, in Stockholm, and were stored in darkness until the analyses were performed. Every sample was analyzed for its content of dyes and approximately every 10th sample was analyzed for the presence of  $I^-$  and  $Br^-$ . This sampling procedure was chosen because of the long time required by the ion-selective analyses which were measured in minutes compared with seconds for the analyses by spectrophotometry. The water samples analyzed by spectrophotometry were buffered to a pH of 8.9. Those samples analyzed with ion-selective electrodes had a  $NaNO_3$  solution added to increase its ionic strength. The added pH buffer was in solid form to eliminate compensation due to dilution of the sample. The composition of the buffer is given in Chapter 4.3. The spectra obtained from the analyses using the spectrophotometer were stored on magnetic disks. These spectra were used in solving for the multi-component system. Each disk can store 150 spectra.

## Equipment

The analyzing equipment consists of three major parts:

- Sample handler
- Spectrophotometer used for dyes
- Ion-selective electrodes for salts.

The sample handler used in laboratory was the same type as the fractional collectors used at the field site but it was rebuilt to handle the sampling of the test tubes.

The computer code, used for the multi-component analysis of the dyes, was transferred from the HP85 to the HP86 and modified to be more flexible in the setup of the analyzing schemes. The modified code allows for the following:

- Selection of which dyes to solve for (up to 7)
- Selection of standards for each dye (up to 10 per dye)
- Selection of which part of the spectrum to use ( $\lambda_{\min}$ ,  $\lambda_{\max}$ )
- Selection of which part of the spectra, measured and calculated, should be plotted (this could be different from  $\lambda_{\min}$  and  $\lambda_{\max}$  used calculating the concentrations)
- Compensation for particles present in the sample.

The automatic system for the analysis of the salt by ion-selective electrodes was modified to allow for the analysis of  $I^-$  and  $Br^-$  at the same time. Since the ion-selective electrodes are sensitive to temperature and could be poisoned from exposure to high concentrations the system performed frequent calibration of the electrodes. The system was controlled by the HP86 computer with which it was possible to set the following:

- Sample volume
- Time allowed for stabilization before actual measurement
- Number of rinsings with next sample when changing samples
- Number of samples measured between calibrations
- Number of calibration solutions and concentrations
- Total number of samples
- Type of ion  $I^-$  or  $Br^-$  or both.

The sample handler for the ion-selective analyzing system is the same as for the spectrophotometer system. In calculating the concentration,

the results from the calibration measurements before and after the actual measurement were used and were weighted in time. The automatically calculated  $I^-$  and  $Br^-$  concentrations were stored on magnetic disks.

## 4 TRACER

### 4.1 INTRODUCTION

To be able to distinguish from which of the nine different injection zones a tracer had been injected, at least nine different tracers had to be used. Abelin et al., (1985) have tested different dyes for their suitability as conservative tracers. Of the tracers used in the single fracture experiment the following were considered for use in the 3-D Migration experiment:

- Uranine
- Iodide
- Eosin
- Elbenyl Brilliant Flavine.

At least 5 more tracers were needed. It was decided that the analyzing methods for the new tracers should be the same as for the old tracers i.e. spectrophotometry and ion-selective electrodes.

### 4.2 REQUIREMENTS

In a fractured porous medium, small tracer molecules that mix in with the flowing water will have access by diffusion to the medium pores and thus will have a velocity less than the water flowing in the fracture. A comparison between the residence times obtained from diffusing and "nondiffusing" tracers show higher water velocities for the case of the "nondiffusing" tracers.

The following was considered in choosing a tracer:

- It should be stable over the time of the experiment
- It should not change the properties of the water being traced
- If several tracers are used, there should not be any interactions between them
- The dynamic range of the response, i.e. solubility in relation to detectability should be high
- There should not be any interactions between the tracer and the surroundings
- The tracers should, if possible, not be hazardous
- The tracer should be stable within the pH range of the water
- The tracer should be stable within the temperature range used (10 - 25 °C)

- The tracer should not decompose due to light exposure.

A survey of suitable dyes was performed and approximately 100 candidate dyes were tested in the laboratory.

#### 4.3

#### THE DIFFERENT LABORATORY TESTS

The laboratory work can be divided into four parts:

- Batch experiments to select suitable conservative tracers  
(First test to select dyes suitable for further testing)
- Extended batch tests of selected tracers  
(Test for sorption and stability)
- Testing of large molecular weight tracer
- Testing of the 7 selected tracers to determine the effect of matrix diffusion.

All batch experiments were performed using ground water collected in the Stripa Mine, with a pH 8.2 - 8.3. In the diffusion experiments water collected at the actual test site was used. The chemical composition of the groundwater is given in Table 4-1.

The test solutions were kept in 200 ml glass flasks. During the laboratory tests, the flasks were stored in a dark place and only retrieved during the water sampling. Samples of 1-2 ml were taken on the average once a week.

Before analyzing the samples, the solutions were buffered to a pH of 8.9. The buffer had the following composition:

3.092 g	H <sub>3</sub> BO <sub>3</sub>
3.728 g	KCl
0.852 g	NaOH

The buffer compounds were added as a dry mixture to eliminate the effect of dilution.

Table 4-1. Ground water composition, 12 samples were taken at different places within the 3-D test site.

Specie	Concentration. [ ppm ]	
	min	max
Al	8 <sup>1</sup>	52 <sup>1</sup>
Ca	18	32
Cl	18	92
F	3.0	3.6
Fe <sup>2+</sup>	<0.01	<0.01
Fe <sub>tot</sub>	0.02	0.97
HCO <sub>3</sub>	18	93
I	<0.01	0.03
K	0.11	0.28
Mg	0.033	0.22
Mn	<0.02	0.05
N (NH <sub>4</sub> )	<0.01	.02
Na	31	60
NO <sub>3</sub>	<0.01	0.16
NO <sub>2</sub>	0.001	0.062
P (PO <sub>4</sub> )	0.02	0.07
Si	9.3	18
SO <sub>4</sub>	1.6	2.8
TOC	1.9	5.0
Conduct.	22.9 <sup>2</sup>	48.7 <sup>2</sup>
pH	7.2	8.2

<sup>1</sup> in ppb

<sup>2</sup> in mS/m

#### 4.4 BATCH EXPERIMENT

To reduce the amount of work there was a first sorting out of dyes which had the following characteristics:

- low solubility
- not stable with time
- low detectability
- diffuse spectrum.

Tests were run for 30 days for four different concentrations, 100, 10, 2, and 1 ppm.

The conceivable tracers were selected from different suppliers of dye stuffs. A table of the tested dye and results are given in Appendix 2.

#### 4.5 EXTENDED BATCH TESTS

Out of the remaining 91 tested dyes, 40 were selected for further testing. For the test the 40 dyes were divided into 10 groups, see Table 4-2. These dyes were tested for the following:

- sorption on granite
- sorption on materials in the equipment
- stability with time.

The dyes in one group were selected in such a way that the wave length for the maximum absorbance did not overlap. The dyes in each group were mixed in a test solution. These tests were run for 320 days.

The granite particles used in the tests were in the size range 1-2 mm. The particles were washed and dried before mixing with the dye solution. The amount of granite particles was 10 g. The following materials were used in testing for sorption on equipment materials: Polystyrene, 5 g, from test tubes; soft PVC, 5 g, from plastic sheets; and clear PVC, 5 g, from tubing. All the materials were cut to small sizes.

Table 4-2. Dyes selected for further tests.

Group	Dye	Rejected <sup>1</sup>	Conc. [ ppm ]
1	Direct Yellow 59	NSwT	2
	Brilliant Yellow 20	SG/SM	2
	Acid Red 94	OK	2
	Duasyn Acid Green V	OK	2
	Procion Turquoise H-A	NSwT	1
2	Uranine	OK	2
	Eosin yellowish	OK	2
	Direct Blue 67	NSwT	1.5
3	Acid Yellow 11	SG/SM	1.5
	Duasyn Acid Yellow xx	SG	2
	Eosin blueish	OK	2
	Phloxine B	OK	1.5
4	Duasyn Acid Yellow RRT	SG	2
	Alphanol Fast Violet FGR	SM	1.5
	Duasyn Acid Blue VF	SG	2
5	Procion Orange MX-2R	NSwT	2
	Acid Green 50	SG/SM/ NSwT	1
6	Reactive Blue 21	SM/NSwT	1.5
	Duasyn Acid Rhodamine -B	SM/NSwT	1.5
	Mordant Yellow 8	NSwT	1.5
	Procion Brown H-3R	NSwT	1.5
	Procion Red HE-3B	NSwT	1.5
7	Procion Green H-E4BD	NSwT	2
	Acid Yellow 17	SG/NSwT	10
	Procion Red H-8BN	SM	10
	Levafix Schwartz E-B	SG/NSwT	10
	PINA Acid Green A anti-halo	SG/SM/ NSwT	10
8	Cibacron Brilliant Green	NSwT	10
	Acid Yellow 36		10
	Solvent Red 72	SG/NSwT	10
	Cibacron Rot F-B	NSwT	10
	Procion Navy H-3R	NSwT	10
	Bromothymol Blue	SG/DtD	10
9	Elbenyl Brilliant Flavine	OK	10
	Procion Red MX-5B	SM	10
	Cibacron Blau TR-E	NSwT	10
	Acid Blue 62	NSwT	10
10	Procion Scarlet MX-G	SM	1.5
	Sumifix Supra Br Red 3BF	SM	1.5
	Elbenyl Blue FBLL	NSwT	2

<sup>1</sup> Major cause of rejection:

DtD Difficult to dissolve  
 NSwT Not stable with time  
 OK Usable as tracer  
 SG Sorption on granite  
 SM Sorption on materials

#### 4.6 LARGE MOLECULAR WEIGHT TRACER

It was also of interest to use a conservative tracer with a low diffusivity, i.e. that would reduce the matrix diffusion and thereby give more directly the flow velocity in the fracture. A total of 7 different large molecular weight tracers (15000) were synthesized and tested. In all the cases, PEG (Polyethylene glycol) was the large molecule to which different chromophore groups had been added. At most two chromophore groups could be added to one large molecule. This low number of chromophore groups makes the sensitivity of the spectrophotometry low and thus a lower dynamic range for the large molecular weight tracer. Table 4-3 shows the 7 synthesized large molecular tracers.

Table 4-3. Synthesized large molecular weight tracers.

Name	Dye	$\lambda_{\max}$	Sorp.Gr.	Stability
STR-1	Procion yellow MX-R	390	none	good
STR-2	Levafix schwartz E-B	590	small	good
STR-3	Procion red HE-3b	510	small	good
STR-4	Direct Yellow 59	340	large	good
STR-5	Brilliant Cresyl Blue	605	difficult to dissolve	
STR-6	Procion Orange H-2R	485	none	good
STR-7	Procion Red H-8BN	545	none	good

The large molecular tracers were tested the same way as were the dyes. Out of the 7 tracers only two were found suitable as tracers. These tracers were STR-6 and STR-7. Tracer STR-7 was chosen before STR-6 because of its  $\lambda_{\max}$  location. In the case of STR-6, its  $\lambda_{\max}$  coincides with that of Uranine.

#### 4.7 SELECTED TRACERS

The selected dyes to be used as tracers are given in Table 4-4 together with the analyzing method and used dynamic range. The spectra for all the suitable dyes are given in Appendix 3.

A material loss of 1/100 of the tracer in the solution due to adsorption could be detected so a dynamic range of 100 was assumed. From these observations, the corresponding sorption coefficient value,  $K_D$ , of  $0.5 \text{ m}^3/\text{m}^3$  was the minimum  $K_D$  that could be detected.

Table 4.4 Dyes selected to be used as tracers.

Dye	Injection conc. ppm	Dynamic <sup>3</sup> x 10 <sup>-3</sup>
Acid Red 97 (Rose Bengal)	1500	5 <sup>4</sup>
Bromide	2000	1 <sup>5</sup>
Duasyn Acid Green	1500	50 <sup>4</sup>
Elbenyl Brilliant Flavine	2000	5 <sup>4</sup>
Eosin blueish	1500	10 <sup>4</sup>
Eosin yellowish	1000	10 <sup>4</sup>
Fluoride <sup>1</sup>	40000	10 <sup>5</sup>
Iodide	2000	5 <sup>5</sup>
Phloxine B	1500	10 <sup>4</sup>
STR-7 <sup>2</sup>	40000	5 <sup>4</sup>
Uranine	1000	100 <sup>4</sup>

- 1 To be used together with STR-7
- 2 Large molecular weight tracer
- 3 Injection concentration divided by detection limit  
(a very rough figure dependent on the mix of tracers)
- 4 Analyzed by spectrophotometry
- 5 Analyzed with ion-selective electrodes.

During the whole experiment blank solutions containing tracers were stored in the laboratory. This was done so to detect any degradation of the tracers during a long time period.

After the final selection of the 7 tracers, each of the tracer was tested separately for stability and sorption on granite for an additional 140 days.

#### 4.8 THROUGH DIFFUSION EXPERIMENT

As mentioned earlier all tracers will more or less penetrate into the porous matrix due to diffusion. From the through diffusion experiment it was not only possible to determine the diffusivity but also to obtain the combined effect of sorption and diffusivity. The laboratory tests gave values of the interaction between the tracer and rock which were put into the Advection-Dispersion-Matrix Diffusion model. For further information on the actual experiments see Report 3, "Performed Experiments, Results and Evaluation."

Table 4-5. Compiled results from the through diffusion experiments.

Tracer	Diffusivity $\text{m}^2/\text{s} * 10^{14}$
Bromide	31
Elbenyl Br. Flavine	1.2 - 7.8
Eosin blueish	1.2 - 4.3
Eosin yellowish	1.5 - 2.9
Fluoride	< 5
Iodide	11 - 29
STR-7	< 0.01
Uranine	0.9 - 1.7

The evaluation of the interaction of the different tracers and the rock shows that there was no difference in this interaction, except for the diffusivity, between the tracers within the resolution range of the experiment.

## 5 DISCUSSIONS AND CONCLUSIONS

### 5.1 GENERAL

The equipment used in the experiment contributed little problems overall. The major problem experienced was the difficulties in maintaining "constant" operating conditions which were independent of changes in the boundary conditions, e.g. holes in the mine were opened and closed during the injection period.

### 5.2 FIELD EQUIPMENT

#### Bentonite packer system

There are two major drawbacks with a bentonite packer system: (1) it is not removable, and (2) there is a very short time period available for installation. There are two major advantages with a bentonite packer system: (1) the sealing improves with time and needs no external power supply; and (2) it is possible to completely seal off the whole length of the hole except for the zones of interest.

If the time required for the bentonite to swell could be prolonged by some coating of the bentonite, the very tight time schedule for installing the system may be prolonged.

#### Tracer injection system

Based on the experience from earlier experiments (Abelin et al., 1985), a modification of the nitrogen supply and pressure regulation was done. However, large variations of the injection flowrates still occurred. These variations were not due to variation of the injection pressure but were caused by the variation of the "natural" pressure at the injection zones. The "natural" pressure variations were induced by the opening and closing of the boreholes adjacent to the test site which were needed by other experimental programs present in the mine. A system designed with a constant injection flowrate would have handled these variations but the injection pressure would have varied instead.

At the start of the experiment, different techniques for injecting tracers with a constant flowrate were tested. The major problem was the low injection flowrates of 1–10 ml/h combined with the groundwater which was not very "clean". In addition, the system was to run continuously for one to two years. Two injection systems were developed which both were based on the displacement of a piston. For each drive unit, two pistons were placed in opposite directions and connected. One piston was used for injection while the second one was refilled and the system would switch automatically between the two pistons. The in and out flows from the pistons were regulated by a 4-way valve.

The first system used a step motor and a linear moving unit as a drive unit for the two pistons. In the second system the linear movement was achieved by a second set of two pistons with diameters

3 times that of the injection pistons. From this arrangement it was possible to use a pump with a flowrate approximately 10 times the injection flowrate and also use oil as a pumping medium to prolong the life of the pump.

It was found that none of the above systems were reliable over long periods of time. It was therefore decided to use the injection method with a constant pressure instead.

#### Water sampling equipment

The sheets were in place for more than two years and only minor problems were experienced. During this time, monthly periodic checking of the sheets were performed, one problem experienced was that small loose rock pieces fell into the sheets and would have to be cut out. Other minor damages to the sheets were fixed with melting glue.

The specially designed flow valve, which was installed to switch to a bottle when the test tube was full, worked very well during the whole test period.

#### Pressure monitoring system

In the pressure monitoring system, the pressure transducers were never in direct contact with the water but had an interface of silicone oil. This oil prolonged the life of the transducer and eliminated contamination with tracer solution.

#### "Ventilation" experiment

In the ventilation experiment the major uncertainty in determining the water balance for the inlet and outlet air was not the variations in the air velocity but the small difference in the temperature and relative humidity between the inlet and the outlet air.

**REFERENCES**

Abelin, H., Gidlund, J.: "Migration in a single fracture. Instrumentation and site description". Stripa Project, Internal report 85-03, Stockholm, 1985.

Pusch, R., Personal communication, 1983.

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- Figure A1-1. Map over the 3-D test site and adjacent areas.
- Figure A3-1. Duasyn acid green.
- Figure A3-2. Elbenyl Br. Flavine.
- Figure A3-3. Eosin blueish.
- Figure A3-4. Eosin yellowish.
- Figure A3-5. Phloxine B.
- Figure A3-6. STR-7.
- Figure A3-7. Acid red 97 (Rose Bengal).
- Figure A3-8. Uranine.

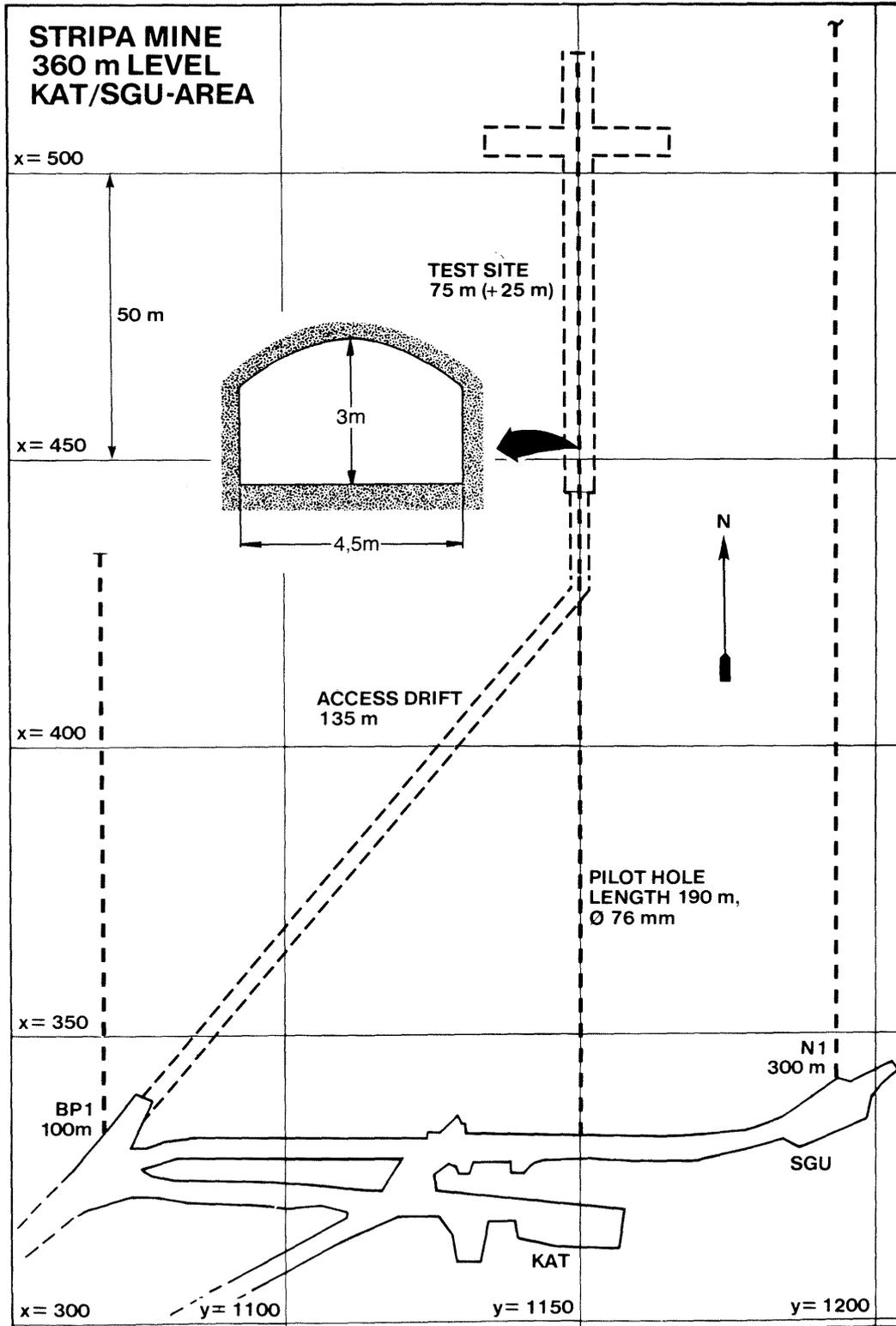


Figure A1-1. Map over the 3-D test site and adjacent areas.

Table A2-1. Dyes selected for the first test.

Name	Color Index	Rejected <sup>4</sup>	Supplier
Acid Blue 62	-	OK	L.B Holliday & Co
Elbenyl Brilliant Blue RN			
Acid Green 1	45410	NSwT	Aldrich
Naphtol Green B			
Acid Green 25	61570	LowEx	L.B.Holliday & Co
Elbenyl Blue Green GNN			
Acid Green 50	44090	OK	Aldrich
Lissamine Green B			
Wool Green S			
Acid Orange 3	-	Dt S	Diazo-kemi <sup>1</sup>
Acid Orange 88	-	BP	Chemapol
Ostalan Orange RL Supra			
Acid Red 94	45440	OK	Aldrich
Rose Bengal			
Tetraiodotetrachlorofluorescein sodium salt			
Acid Red 97	-	Dt D	Chemapol
Alizarine Chrome Red G			
Acid Red 213	-	BP	Chemapol
Ostalan Bordeaux FGRL			

Table A2-1 continued.

Acid Yellow 11	18820	OK	Aldrich
Fast Acid Yellow			
Flavazin L			
Flavazine			
Zapon Fast Yellow CGG			
Acid Yellow 17	18965	OK	Aldrich
Acid Yellow 36	13065	OK	Aldrich
Metanil Yellow			
Acid Yellow 117	-	DtD	Dept. Org.Chem <sup>2</sup>
Alphanol Fast Violet FGR	-	OK	Hoechst
Amido Flavin FFP ( EA AC 005 )	-	BP	Hoechst
Brilliant Yellow 20, 137-5	-	OK	Dept. Org.Chem <sup>2</sup>
Bromothymol Blue	-	OK	Janssen
Cibacron Blau 3G-A	-	LowEx	Dept. Org.Chem <sup>3</sup>
Cibacron Blau F-R	-	BP	"
Cibacron Blau TR-E	-	OK	"
Cibacron Brilliant Green T3G-E	-	OK	"
Cibacron Gelb F-3R	-	BP	"
Cibacron Gelb F-G	-	BP	"
Cibacron Rot F-B	-	OK	"
Direct Blue 67	-	OK	Chemapol
Saturn Blue L3R			
Direct Blue 78	-	BP	Chemapol

Table A2-1 continued.

Direct Blue 86	-	LowEx	Chemapol
Saturn Turquoise Blue LG			
Direct Blue 106	-	LowEx	Chemapol
Saturn Pure Blue LB			
Direct Orange 26	-	BP	Chemapol
Direct Fast Orange S			
Direct Orange 39	-	BP	Chemapol
Saturn Orange L7G			
Direct Red 23	29160	NSwT	Chemapol
Direct Fast Scarlet 4B			
Direct Red 76	-	DtD	Chemapol
Saturn Scarlet LGG			
Direct Red 80	-	DtD	Diazo-kemi <sup>1</sup>
Saturn Red F3B			
Direct Yellow 4	24890	OK	Aldrich
Direct Yellow 11	-	DtD	Chemapol
Direct Yellow 12	24895	BP	Aldrich
Chrystophenine			
Chrystophenine G			
Direct Yellow 29	19556	BP	Chemapol
Saturn Yellow LRT			
Direct Yellow 44	-	DtD	Chemapol
Saturn Yellow L4G			

Table A2-1 continued.

Direct Yellow 59	49000	OK	Aldrich
Primulin			
Direct Yellow 62	36900	DtD	Aldrich
Dyasyn Acid Blue VF (DP DR 505)	-	OK	Hoechst
Duasyne Acid Green V (DP AF 605)	-	OK	Hoechst
Duasyne Acid Rhodamine B (DP BB 305 )	-	OK	Hoechst
Duasyne Acid Yellow RRT (DP AF 005)	-	OK	Hoechst
Duasyne Acid Yellow XX (DB BL 005/A)	-	OK	Hoechst
Elbenyl Brilliant Flavine A-FF	-	OK	L.B.Holliday & Co
Elbenyl Blue FBLL	-	OK	L.B.Holliday & Co
Eosin Bluish	-	OK	Merck
Eosin Yellowish	-	OK	Merck
Indigo Carmine	-	NSwT	Aldrich
5.5'-Indigodisulfonic acid disodium salt			
Ink Blue R (DP DZ 505)	-	NSwT	Hoechst
Levafix Brilliant Rot E-2B	-	BP	Dept. Org.Chem <sup>2</sup>
Levafix Brilliant Rot P-3B	-	BP	"
Levafix Schwartz E-B	-	OK	"
Mordant Blue 13	-	BP	Chemapol
Alizarine Chrome Blue B			
Mordant Yellow 1	14025	DtD	Aldrich
Alizarin Yellow GG			

Table A2-1 continued.

Mordant Yellow 8	-	OK	Chemapol
Alizarine Chrome Fast Yellow R			
Naphtmochrome Green	44530	LowEx	Aldrich
Phloxine B	45410	OK	Aldrich
Magdala Red			
Eosin			
Blue Shade 2B			
PINA Acid Green anti-halo	-	NSwT	Riedel-Haen
PINA Acid Green A anti-halo	-	OK	Riedel-Haen
PINA Oxonol Yellow K anti-halo	-	NSwT	Riedel-Haen
PINA Oxonol Yellow N anti-halo	-	NSwT	Riedel-Haen
PINA Acid Violet 520 A anti-halo	-	NSwT	Riedel-Haen
Procion Blue H-B	-	LowEx	Dept. Org.Chem <sup>2</sup>
Procion Blue HE-GN	-	NSwT	"
Procion Blue MX-2G	-	BP	"
Procion Blue MX-3G	-	LowEx	"
Procion Brown H-3R	-	OK	"
Procion Brown MX-GRN	-	LowEx	"
Procion Green H-E4BD	-	OK	"
Procion Navy H-3R	-	OK	"
Procion Navy H-ER	-	LowEx	"
Procion Orange MX-G	-	NSwT	"
Procion Orange MX-2R	-	OK	"
Procion Red H-8BN	-	OK	"

Table A2-1 continued.

Procion Red HE-3B	-	OK	"
Procion Red MX-5B	-	OK	"
Procion Scarlet MX-G	-	OK	"
Procion Turquoise H-A	-	OK	"
Procion Yellow H-3R	-	BP	"
Procion Yellow HE-4R Grans.	-	Dt D	"
Procion Yellow MX-R	-	NSwT	"
Procion Yellow MX-4R	-	NSwT/Dt D	"
Reactive Blue 19	-	BP	Diazo-kemi <sup>1</sup>
Sumifix Brilliant Blau R SP			
Reactive Blue 21	-	OK	Diazo-kemi <sup>1</sup>
Sumifix Turkis Blue G			
Reactive Yellow 116	-	BP	Diazo-kemi <sup>1</sup>
Sumifix Supra Yellow 3RF			
Solvent Red 72	45370.1	OK	Aldrich
4,5-Dibromofluorescein			

Table A2-1 continued.

Sumifix Supra Brilliant Red 3 BF	-	OK	Diazo-kemi <sup>1</sup>
Sumifix Supra Brilliant Red 2 BF	-	BP	Diazo-kemi <sup>1</sup>
Uranine A	-	OK	BASF

<sup>1</sup> Maker not known, bought from Diazo-kemi Sweden

<sup>2</sup> Maker not known, small samples from Dept. of Organic Chemistry, KTH

<sup>3</sup> Probably Ciba-Geigy, small samples from Dept. of Organic Chemistry, KTH

<sup>4</sup> Major cause of rejection:

BP	Broad peak at $\lambda_{\max}$
DtD	Difficult to dissolve
LowEx	Low extinction
NSwT	Not stable with time
OK	Suitable for further tests

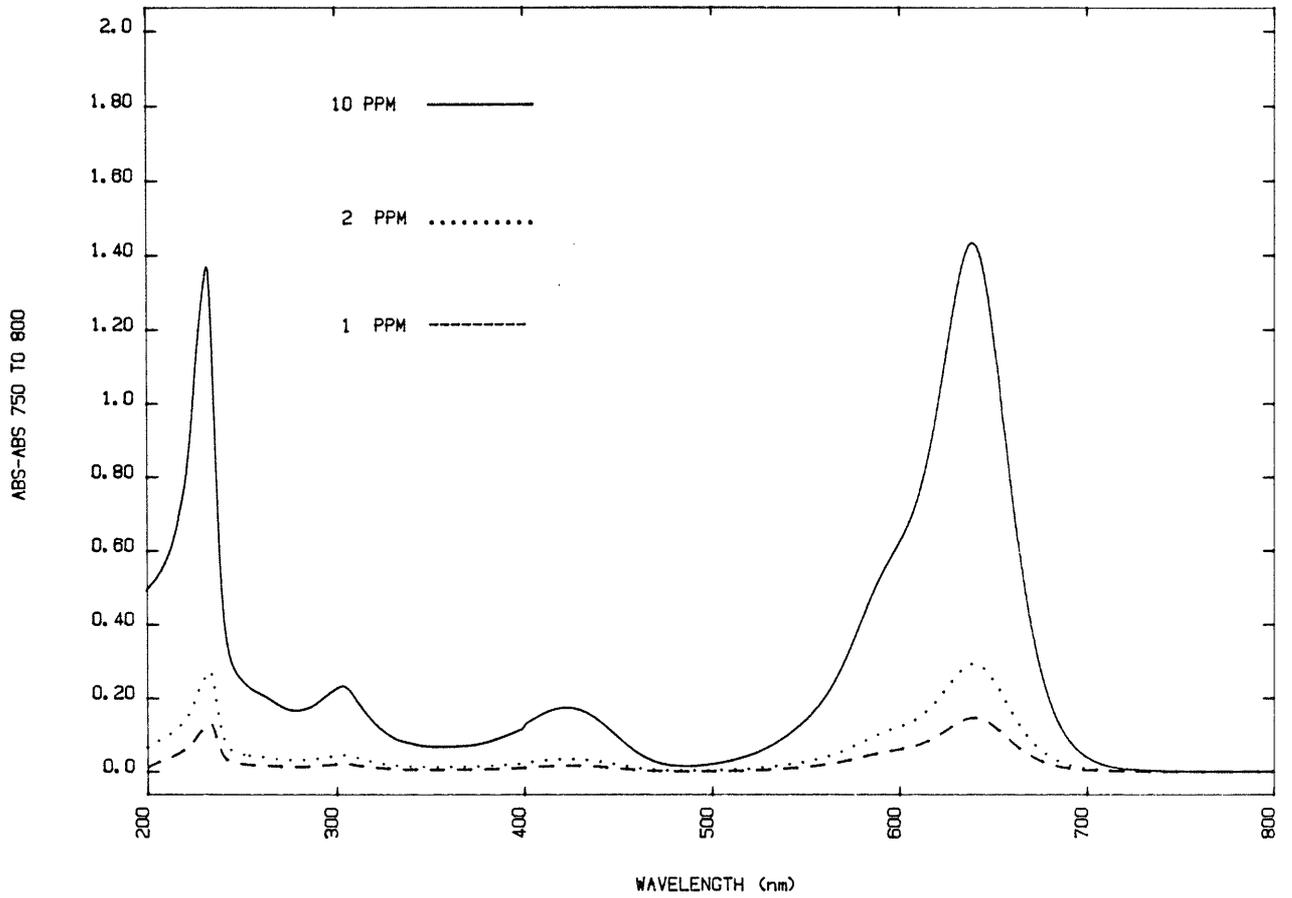


Figure A3-1. Duasyn acid green.

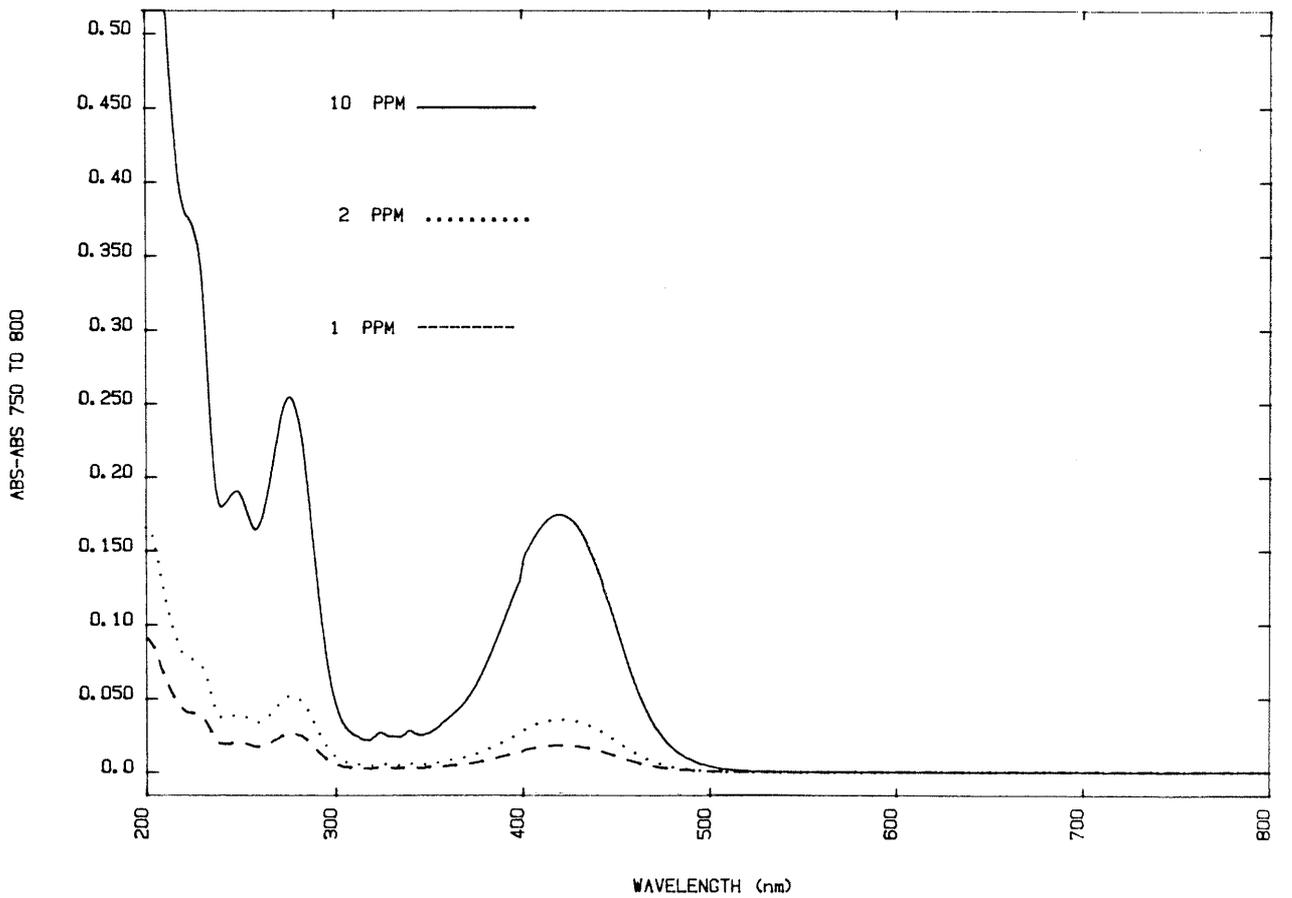


Figure A3-2. Elbenyl Br. Flavine.

A3.2

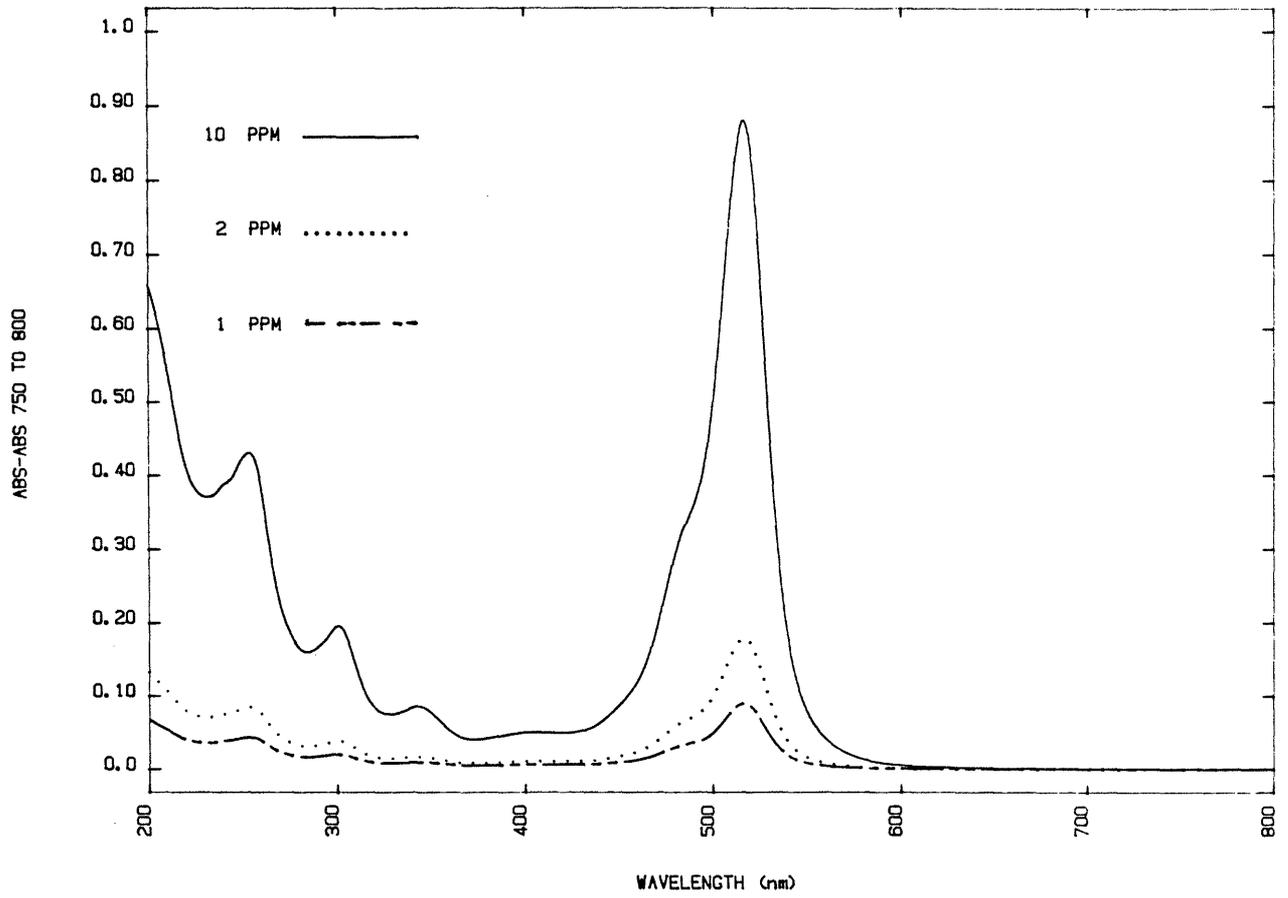


Figure A3-3. Eosin blueish.

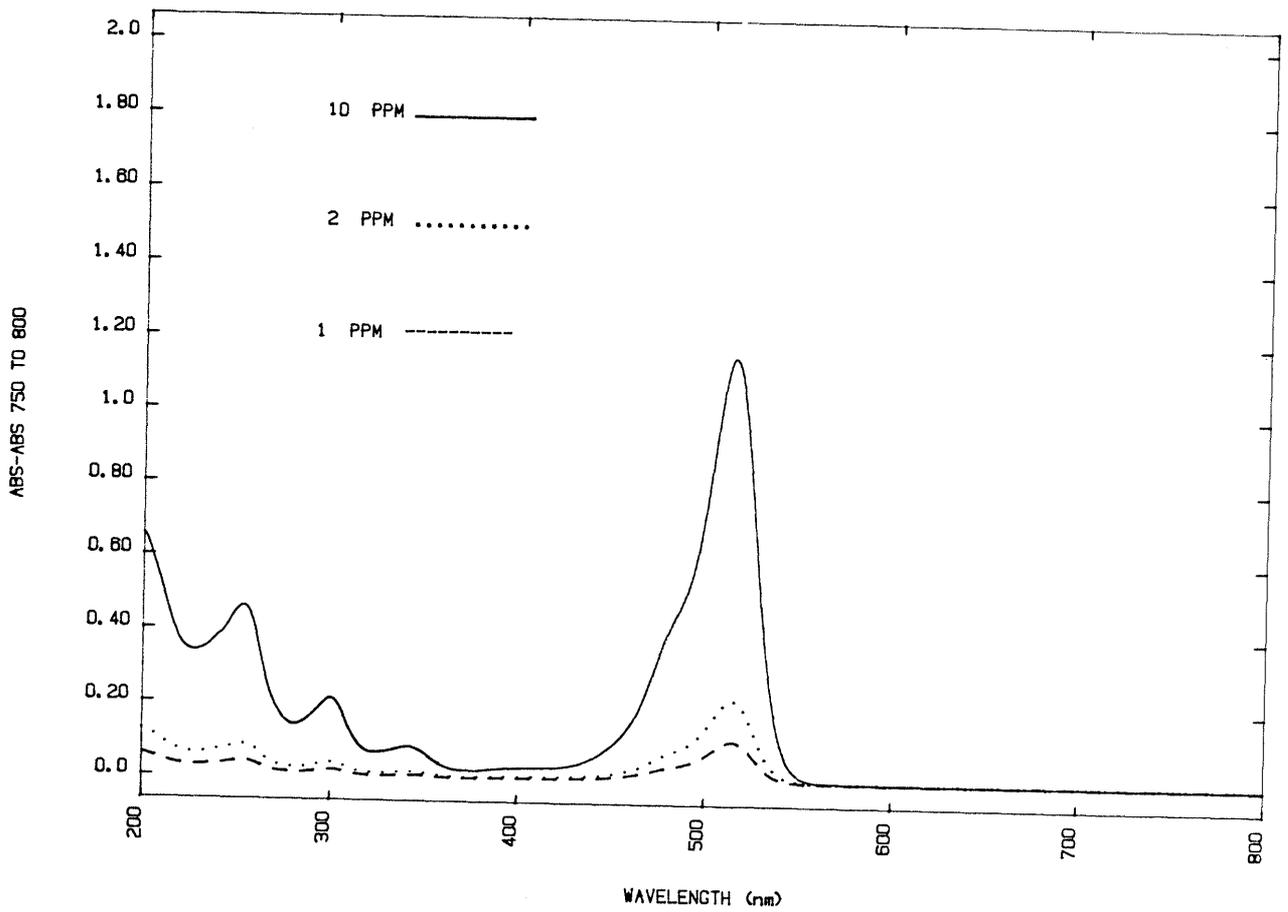


Figure A3-4. Eosin yellowish.

A3.3

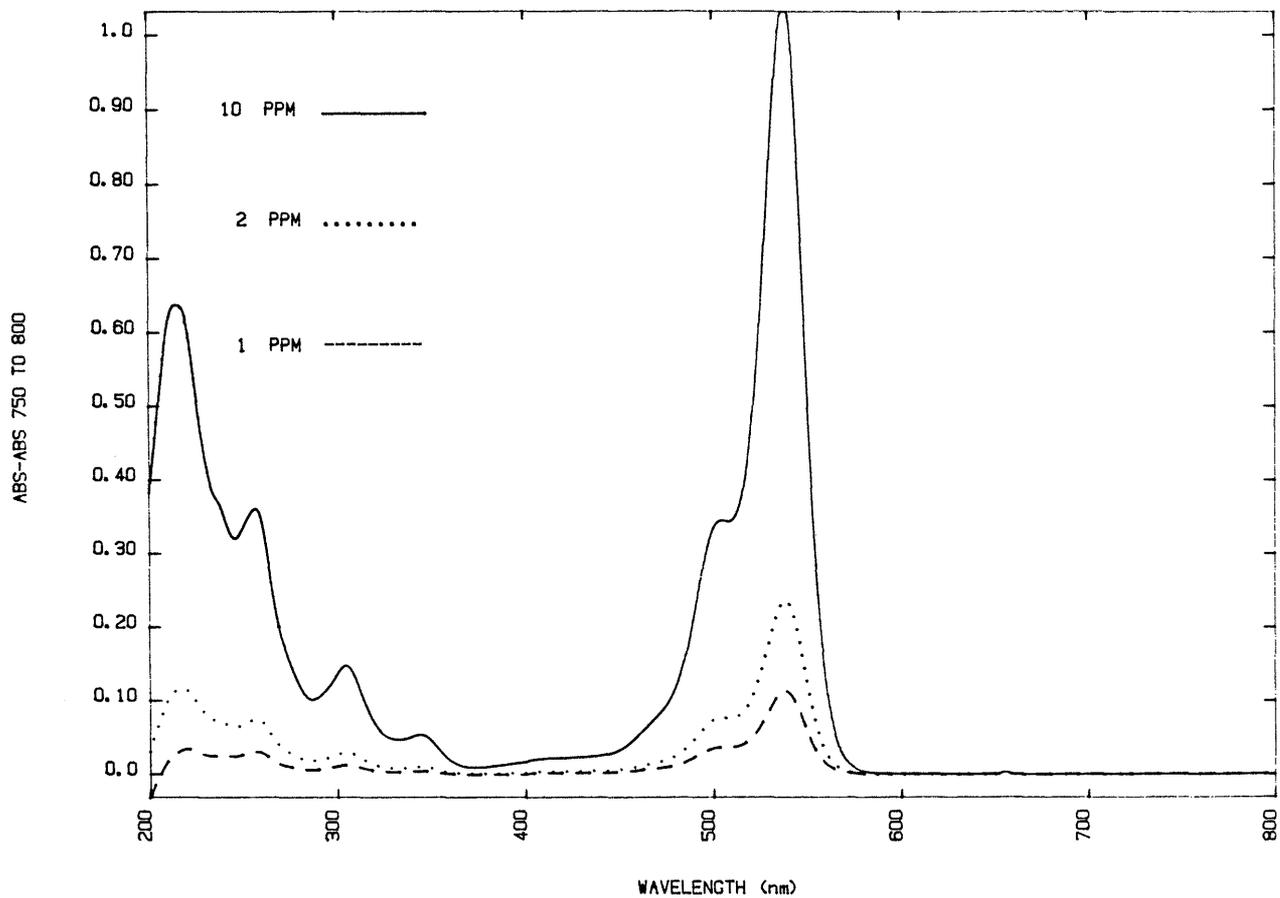


Figure A3-5. Phloxine B.

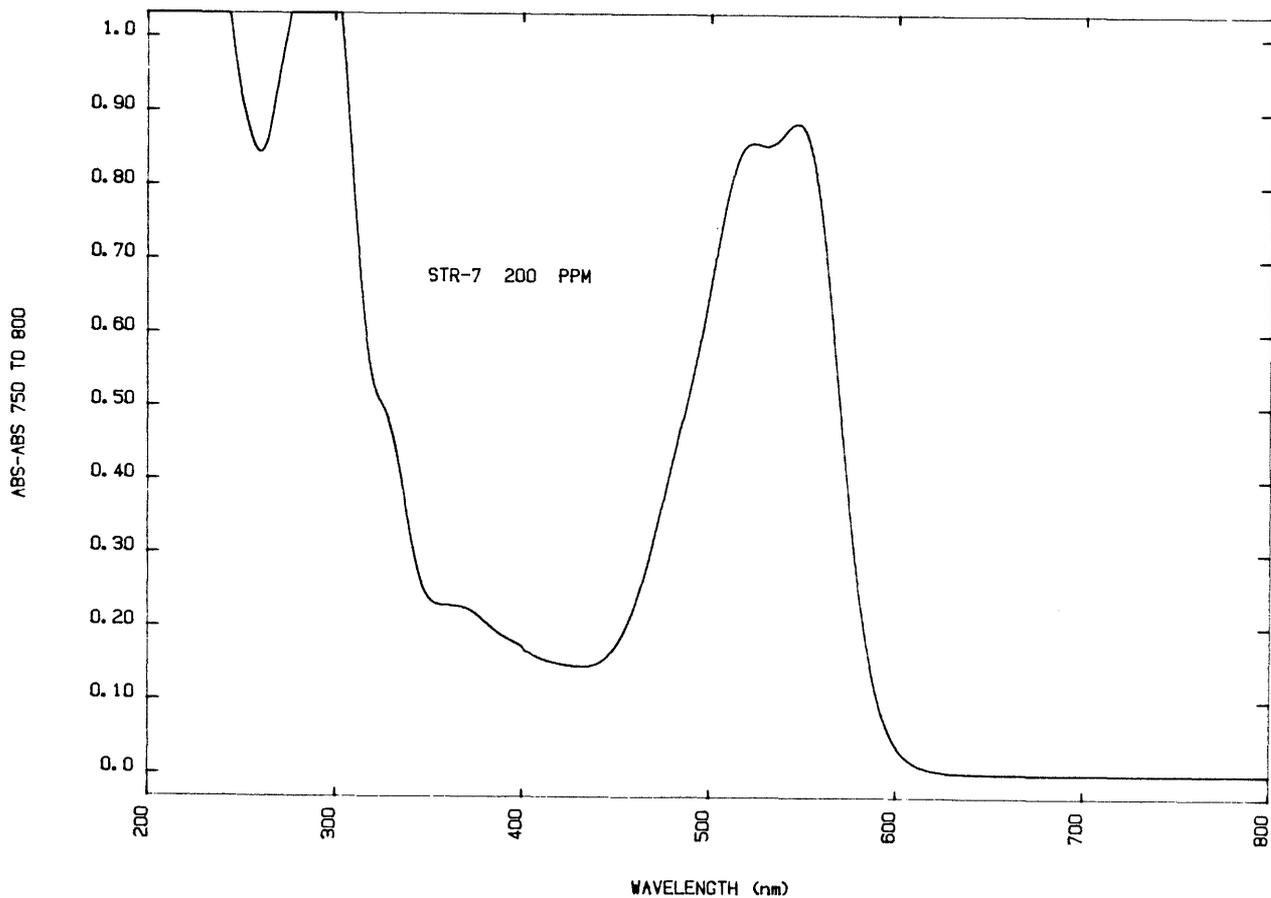


Figure A3-6. STR-7.

A3.4

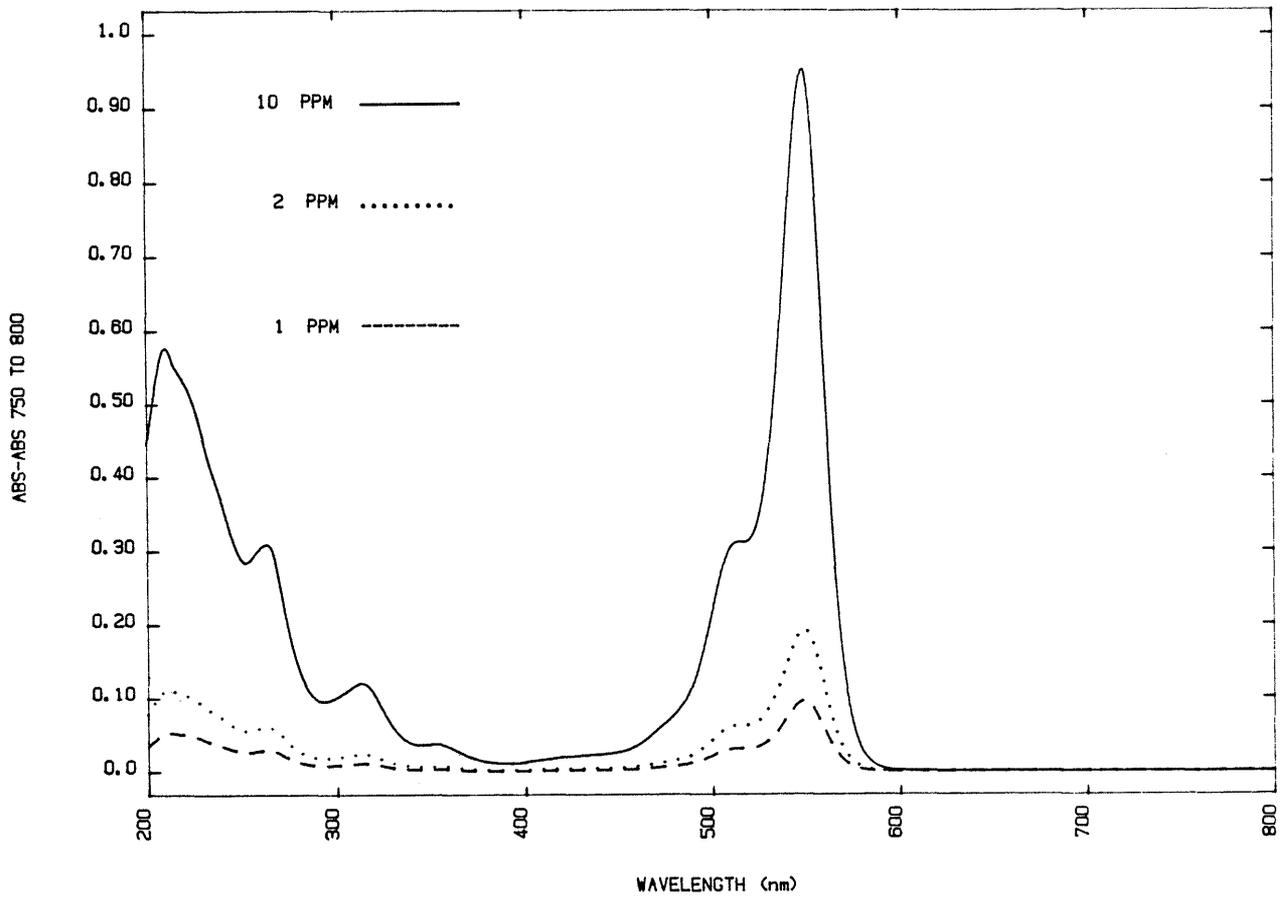


Figure A3-7. Acid red 97 (Rose Bengal).

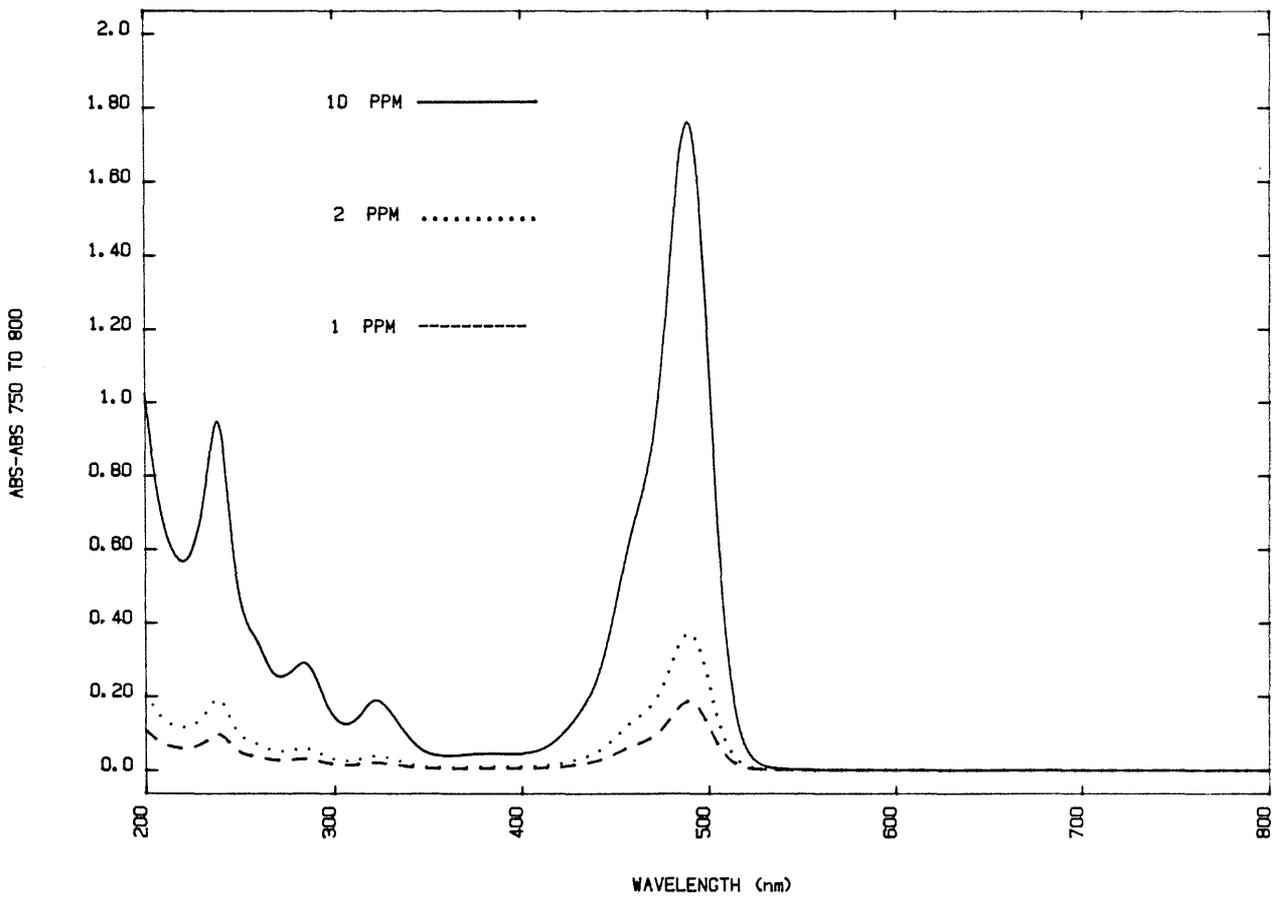


Figure A3-8. Uranine.