

Large Area Solar Cells from Lunar Materials

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Abstract

The first goal of the project has been to produce polymers from materials available on the Moon. This, apparently simple, aim is made complicated by the fact that there is no carbon on the Moon and there are no polymers (with a couple of irrelevant exceptions) known which do not contain carbon. Because of the abundance of silicon and oxygen in the lunar regolith, we decided to aim to produce siloxane polymers with the (-Si-O-) backbone found in silicones. A univalent side group is also needed but there are no univalent elements available in the regolith which could plausibly make stable structures. Failing this, hydrogen is a good choice for side group since it accounts for a small fraction of the total weight of the polymer. Thus we have a group of target structures such as $(-\text{SiH}_2\text{-O})_n$, $(-\text{Si}(\text{OH})_2\text{-O})_n$.

We have approached this goal via a series of simpler syntheses. During the first year we made polydimethylsiloxane $(-\text{Si}(\text{CH}_3)_2\text{-O})_n$ by controlled hydrolysis of $\text{SiCl}_2(\text{CH}_3)_2$, which is a routine synthesis, and then proceeded to attempt to make polydichlorosiloxane by controlled hydrolysis of SiCl_4 . At the end of the first year we had some infra-red spectra indicating that this product had been obtained.



Polydichlorosiloxane

We now have made gram samples of a yellow-brown rubbery polymer which we believe to be polydichlorosiloxane. This material has remained rubbery for over 6 months, sealed in a glass tube. On exposure to air it slowly converts to a powder, presumably silica. We are working to obtain clearer spectral data on this material. Since it has not been previously reported we have no comparative data to establish the structure.

The decomposition behavior of this material is both pleasing and disappointing. A concern with this polymer is that it might disproportionate, that is convert to silica, SiO_2 , and silicon tetrachloride. Such a reaction could be driven by the very high stability of silica. This apparently does not happen. We also expected the polymer to be very sensitive to hydrolysis, which seems to be the case. We had hoped that it might be self-passivating by forming an impermeable layer of silica which would then prevent further reaction.

Polydihydrosiloxane

We are now in the process of trying to make this polymer by hydrolysis of dichlorosilane SiH_2Cl_2 . This synthesis is rather more tricky since the silane is a gas and is expected to be spontaneously inflammable. A vacuum line has been set up to bubble the silane into tetrahydrofuran containing a small amount of water. After a known amount of gas has been passed into the solution, the solvent is evaporated. So far traces of oily residue have been observed but it is not clear how polymeric this is.

Future directions

The fact that neither the dihydrosiloxane nor the dichlorosiloxane have been prepared previously can be attributed to the expected instability of the materials. As explained above, it may turn out to be self-passivating against oxidation. Disproportionation is also a possibility but there are standard strategies for preventing this in polymers.

The dihydrosiloxane polymer should be the least hindered of any chain molecule so far prepared. This would imply that it should be a very low temperature elastomer. We anticipate using a sol-gel reaction to reinforce the material as necessary to form a series of composites with properties from very flexible up to as stiff as normal reinforced polymers. We envisage this family of materials as fulfilling a range of structural and protective roles in the lunar environment.

PREPARATIVE DETAILS

POLYDICHLOROSILOXANE

Polydichlorosiloxane is formed by the hydrolysis of silicon tetrachloride (SiCl_4) with distilled water. The reaction must be run at a controlled slow rate and the reactants cannot be introduced to each other in the concentrated form. The silicon tetrachloride was diluted by a common solvent, tetrahydrofuran (THF), and the distilled water was diluted by evaporation into a heated nitrogen stream. If these steps to dilute the reactants are not taken and the reactants are mixed at full concentration a violent reaction occurs giving the products HCl gas and silica.

The apparatus for the preparation consisted of a sealed reaction flask, cooled to 0-5 degrees C, that had a continuous flow of dried nitrogen gas. The reaction flask exhaust stream was run through a condenser. The condensed solvents were collected in a second flask sitting in an ice-water bath.

The solvent, 250 ml THF, was placed in the reaction flask and purged with nitrogen gas for about 10 minutes. The first reactant (38 ml SiCl_4) was added to the solvent and stirred continuously throughout the entire reaction by a magnetic stir bar. The second reactant, 5.5 ml distilled water, was introduced by evaporation into the heated nitrogen stream (about 60 degrees C). Stoichiometry dictated that 6 ml of distilled water be used but apparently some of the SiCl_4 was evaporating before the water could be fully introduced. Therefore, we reduced the amount of water by 0.5 ml and the reaction has yielded much more product. The first reaction that was done in this manner had the nitrogen stream at room temperature. This reaction was complete in 47 hours. The nitrogen stream for the second procedure was heated to 40 degrees C, and took 12.5 hours to complete. Total time for complete evaporation for the third and latest procedure was 7.5 hours. The completed reaction was then distilled to remove the remaining solvent. Distillation took an average of 15 hours at 50-55 degrees C. The remaining 10-15 ml of material in the latest procedure was colorless and appeared to have a consistency like that of table jelly at room temperature.

The second reaction produced a product that was a brown color. The reason for this brown color may be that the distillation temperature was too high or that the reagents used were not pure. This is the substance that has been tested to a limited degree. The third reaction product was colorless and tests are about to begin on this sample.

Several attempts have been made to get an accurate FTIR spectrum of the polymer. However, due to the sensitivity of the substance to water in the air the spectra are difficult to obtain. An FTIR spectrum of polydichlorosiloxane in THF overlaid with that of the solvent is attached (*Figure 1*). The peaks of interest are those present in the sample but not in the solvent. A clear peak is seen at around 1100 cm^{-1} which corresponds to the Si-O vibration. In addition there are peaks in the $600\text{-}800\text{ cm}^{-1}$ region which we believe are due to Si-Cl. Silicon tetrachloride has a single peak at 760 cm^{-1} while the polymer has peaks at 614 cm^{-1} and 700 cm^{-1} .

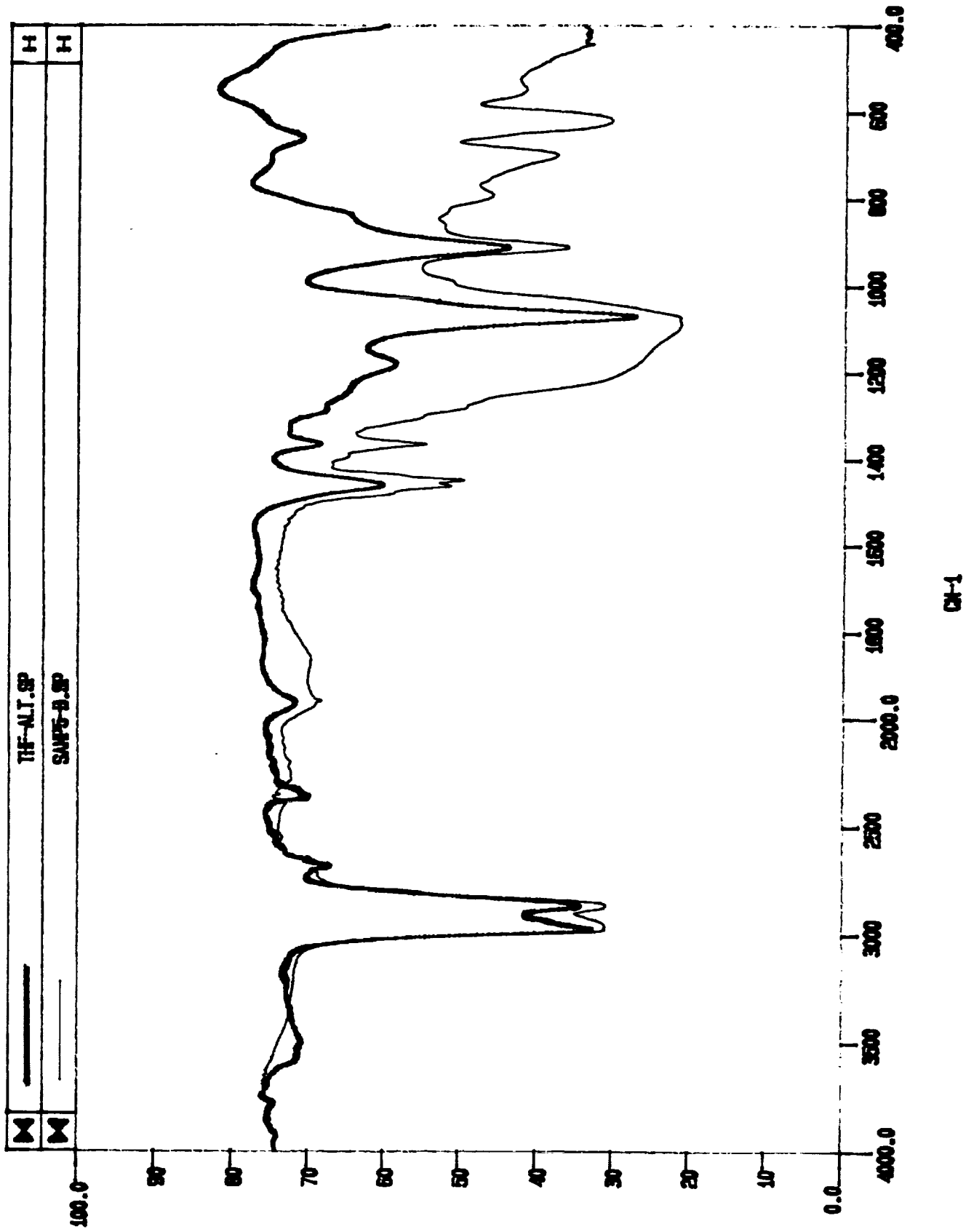
POLYDIHYDROSILOXANE

Polydihydrosiloxane is being synthesized by the hydrolysis of dichlorosilane gas (SiH_2Cl_2). This reaction is being carried out in a vacuum manifold at 1 Torr where the gas is bubbled into a water-THF solution, typically 3 ml water and approximately 140 ml THF. This reaction is being run at room temperature and at reaction times that vary from 30 minutes to 2 hours. After the reaction is determined to be complete, the gas is turned off and the THF is pulled off in the vacuum line and collected in a cold trap. This setup is shown in the attached diagram, *Figure 2*.

The products that have been given off are usually clear light-yellow liquids. If allowed to sit for several days strong fumes are given off and the liquid solidifies without any noticeable visual change. This solid has apparent elastic properties but when prodded with a spatula comes apart like very fine wet sand. This type of product was manufactured several times, but on one occasion a white cloudy substance with a dark brown top layer was noticed. This change happened without explanation.

FTIR spectra from the initial experimental runs show the expected bonds (Si-H and Si-O-Si) although it is difficult to remove all traces of solvent without oxidizing the polymer. The lower spectrum in *Figure 3* shows SiH peaks at 2200 cm^{-1} and SiO peaks at around 1100 cm^{-1} . There is also a substantial amount of residual water which appears as a broad peak at 3400 cm^{-1} and some solvent which is seen as C-H at 3000 cm^{-1} . On exposure to the air to remove the final amount of solvent, it can be seen from the upper curve that there is also some oxidation of the polymer with loss of the SiH and a change in the SiO peak as silica is formed. What we cannot be sure of as yet is how far the polymerization has continued and to what extent we have low molecular weight hydroxysilanes.

FIGURE 1



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REACTION APPARATUS FOR POLYDIHYDROSILOXANE

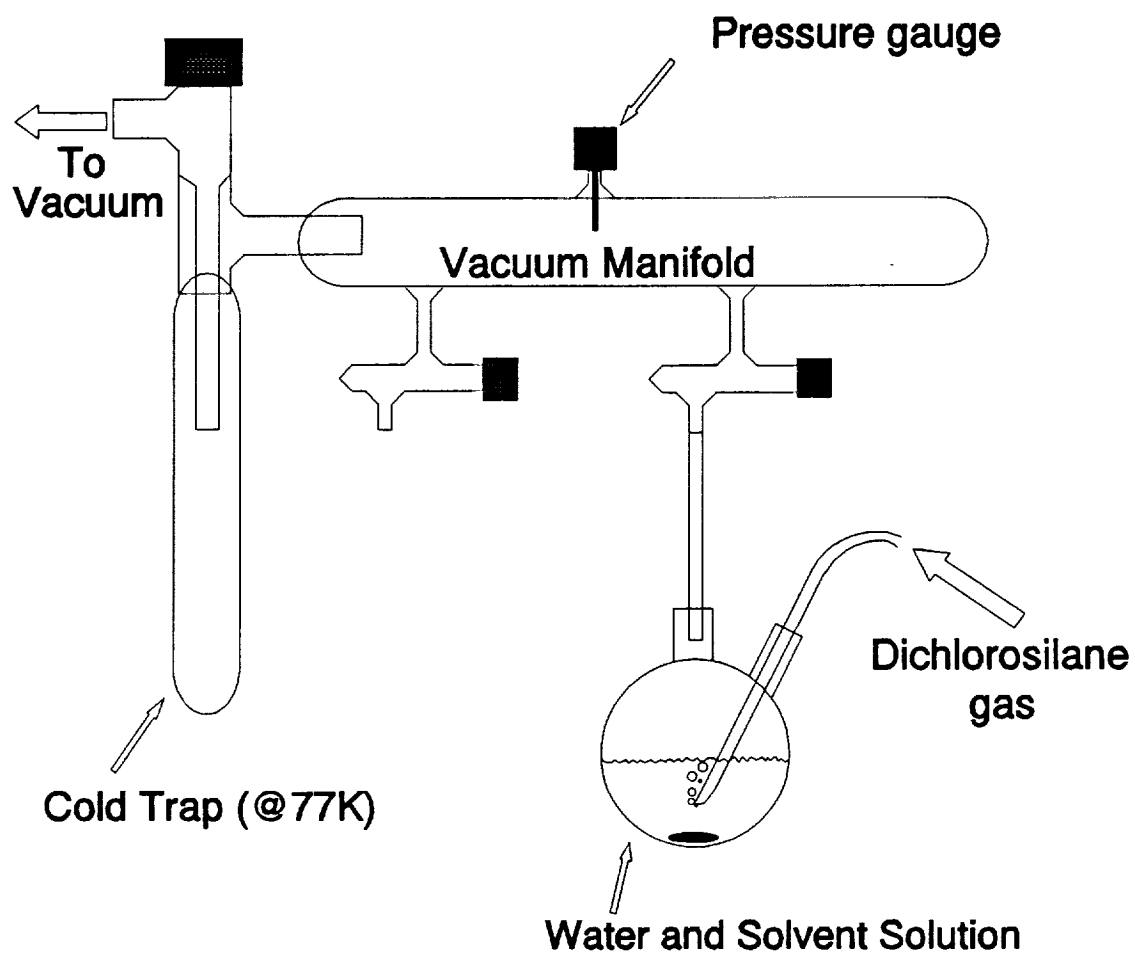
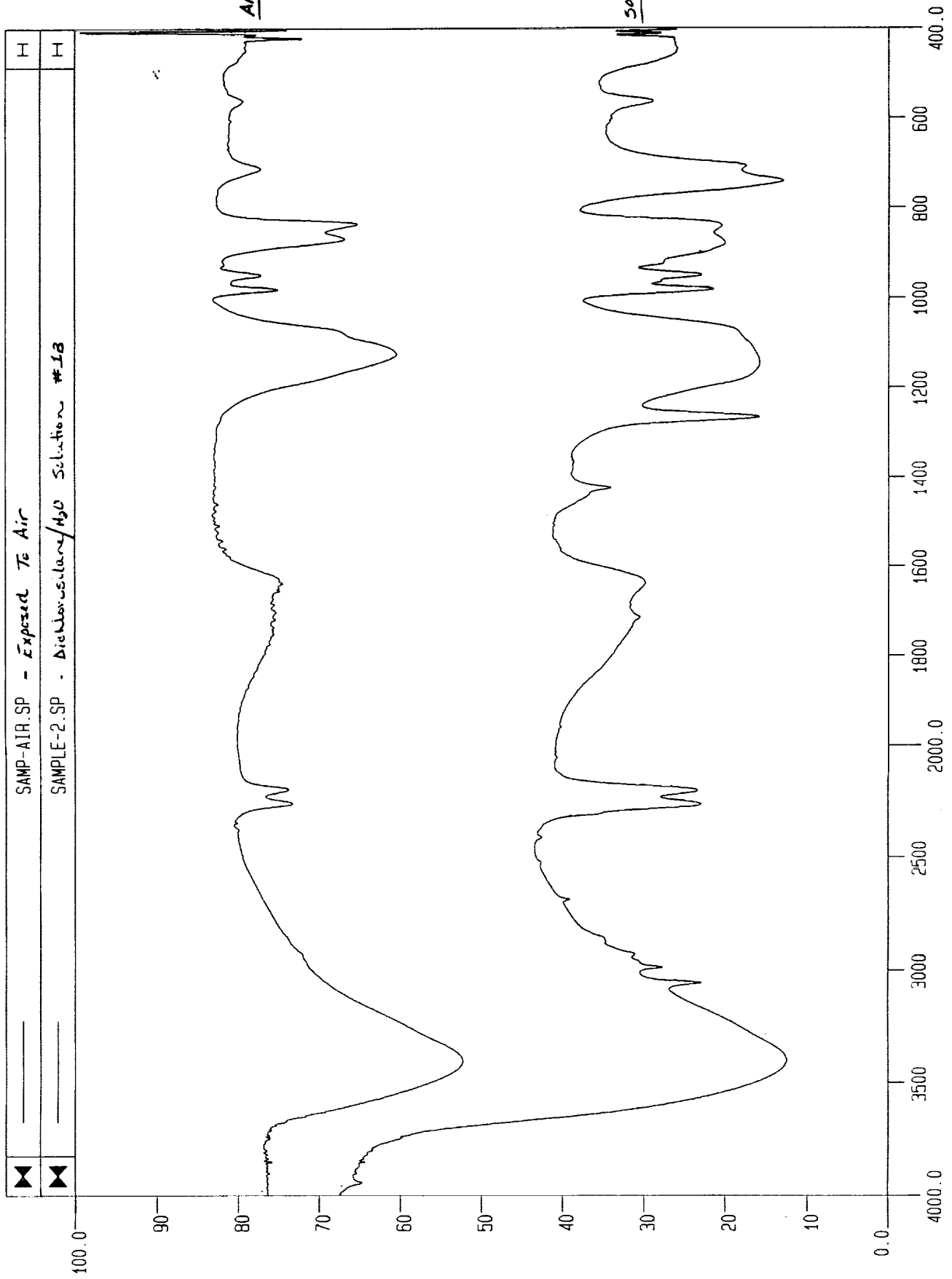


FIGURE 2

FIGURE 3

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