Crystal growth and Liesegang rings

Crystal growth is an area where there has been considerable research. This has been carried out because crystals are useful in the area of solid-state science and, more importantly, they are fun to grow and beautiful to look at!

We recently decided to look at this interesting area of chemistry because of a number of enquiries that we had received about the Advanced Higher Chemistry starter investigation – Crystals[1]. The method of growing crystals described in this starter investigation is the diffusion technique, which often results in the formation of bands of crystals known as Liesegang rings. These are named after the German colloid chemist Dr. R. Liesegang who first described them.

The enquiries that we received centred around problems associated with the preparation of the gels and with the formation of the Liesegang rings.

Gel preparation[2]

The starter investigation describes two methods of gel preparation:

a) Sodium silicate gel[3]

The density of the sodium silicate solution has a direct influence on the pH of the final gel[4] which, in turn, can affect the setting of the gel and ring formation. The starter investigation states that a solution of sodium silicate of density 1.06 g cm^{-3} should be used. We found that it was worthwhile checking the density of the water to be used in the preparation of the solution in order to get this right and to check the density of the solution after making it up before use. A freshly made solution should always be carefully made up and used straight away as results are badly affected by small amounts of contamination.

Several problems can occur when adding the inner electrolyte and the ethanoic acid to the silicate solution. We found the easiest and fastest way was to dissolve the inner electrolyte[2] in the ethanoic acid and then add the silicate solution to the ethanoic acid drop by drop. Adding the ethanoic acid to the silicate solution frequently results in instantaneous setting as well as local ion formation and gel pockets. When adding the silicate solution to the ethanoic acid, we recommend drop by drop addition, using a magnetic stirrer to ensure complete mixing. Drop by drop addition is laborious, time consuming and requires patience but it is necessary to prevent localised ion formation and gel pocket formation.

b) Using a packet of gelatine

The starter investigation suggests, as an alternative to the use of sodium silicate gel, using packets of gelatine to prepare gelatine gels. We used the method suggested in the Schibeci and Carlson paper[5] – "Add gelatine (1.5 g) and one of the chemicals (2.5 g) to 50 ml of distilled water. Heat with stirring until the solution is clear. Pour into a standard test tube (25 mm x 150 mm) until it is about two-thirds full. Stopper the tube and leave to set; this usually takes about 12 h. Add the second solution above the gel nearly to fill the test tube. Do not stopper, but cover with a watch glass" - and found that this gel was very easy to prepare.

For both systems the gels were left to set overnight before the addition of the outer electrolyte. At the setting stage it is very important to ensure that the test tubes containing the gel are held vertical and kept motionless[4] Diffusion and ring formation reflect the starting contour of the gel surface and an uneven surface leads to uneven diffusion and uneven ring patterns making them difficult to observe and categorise.

Systems listed as having produced Liesegang rings

The gel systems used by us were those suggested in the starter investigation and are listed in the table below.

Gel with Inner Electrolyte	Outer Electrolyte Solution	
cobalt(II) chloride	conc. ammonia	0.880
magnesium chloride	sodium hydroxide	19 mol l^{-1}
magnesium chloride	conc. ammonia	0.880
copper(II) sulphate	silver nitrate	0.1 mol l^{-1}
manganese(II) chloride	conc. ammonia	0.880
copper(II) chloride	sodium hydroxide	19 mol l^{-1}
potassium chromate	silver nitrate	0.1 mol l^{-1}

Table 1 *Gel systems*

Observations & comments

Gelatine

The gelatine is much easier to make up than the sodium silicate gel. Well defined ring systems were obtained, within two weeks, when using gelatine with cobalt(II) chloride/0.880 ammonia (fig.1), copper(II) sulphate/silver nitrate solution and magnesium chloride/0.880 ammonia (fig.2), manganese(II) chloride/0.880 ammonia and potassium chromate/ silver nitrate solution (fig.3).

The systems using 19 mol l^{-1} sodium hydroxide produced poor results as the hydroxide flooded through the tube very quickly and the rings formed were very close together and indistinct. Apart from the poor results, we have concerns regarding the use of sodium hydroxide at this concentration. Thinking of the COSHH requirement to, whenever

Fig.1 cobalt chloride + conc. ammonia

possible, substitute a hazardous substance with a less hazardous alternative we tried 4 mol l^{-1} and 2 mol l^{-1} sodium hydroxide. This resulted in slightly better ring formation although they took some three to four weeks to develop. See Table 2 (below).

Gels	Ring Formation
cobalt(II) chloride/ conc. ammonia gelatine	Good clear ring formation.
magnesium chloride/ sodium hydroxide	Poor electrolytes, clump at bottom of tube.
magnesium chloride/ conc. ammonia	Good ring formation.
copper(II) sulphate/ silver nitrate	Good ring formation, black colour makes some areas difficult to see.
manganese(II) chloride/ conc. ammonia	Good ring formation.
copper(II) chloride/ sodium hydroxide	Poor. Alkali floods through system making it difficult to observe rings brown patches in dark blue.
potassium chromate/ silver nitrate	Strong band at top with very faint rings.

Table 2 *- Gelatine systems*

Sodium silicate

Single bands were formed in the silicate gels but no ring systems developed. Small individual crystals were observed and a gradation of colour along the tube showing the diffusion path of the outer electrolyte could be seen (Fig. 4 & Table 3).

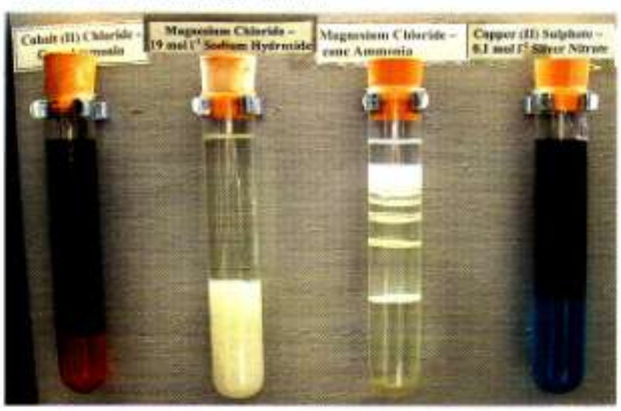
Those systems using 19 mol l^{-1} failed to produce anything resembling Liesegang rings or crystals. Again the sodium hydroxide systems were rerun using 4 mol l^{-1} and 2 mol l^{-1} alkali. This produced better results with some bands being observed. In some cases the use of light and a magnifying glass aided observation of the Liesegang rings and individual crystals.

Silicate gels	Crystal Formation
cobalt(II) chloride/ conc. ammonia gelatine	Strong band at top, colour diffusion down tube.
magnesium chloride/ sodium hydroxide	Poor, nothing discernible.
magnesium chloride/ conc. ammonia	Poor, difficult to discern bands.
copper(II) sulphate/ silver nitrate	Single black band on surface of gel, crystals formed on surface
manganese(II) chloride/ conc. ammonia	Dark band with beige colour diffusing down gel, no crystals observed
copper(II) chloride/ sodium hydroxide	Poor, dark blue colour diffuses through pale copper chloride. No banding pattern identified, impossible to categorise.
potassium chromate/ silver nitrate	Single band formed, crystals formed on band.

Table 3 *- Sodium silicate gel systems*

Safety advice

The safety advice given as part of the Introduction to the Starter Investigations Guide points out that "it is the responsibilty of the teacher/lecturer to ensure that employers' guidelines in relation to the COSHH Regulations are complied with". Whilst this is true, there are many other Regulations that require the employer to assess the risks in the workplace.



Figs.2 & 3 *Gelatine-based gels after about 8 days (Note - bungs were used for diplay purposes only)*

Before work starts on a project it is very important that consideration is given to **any** hazards that could cause serious harm. The process of risk assessment should not be overcomplicated. The HSE expect risk assessments to be suitable and sufficient, not perfect. Following the process of risk assessment it should be possible to demonstrate that:

- a thorough check was carried out
- those who could be affected were consulted
- the obvious significant hazards have been considered and dealt with
- safety precautions have been put in place and that any remaining risk is acceptably low

Fig.4 *Sodium silicate-based gels after about 8 days*

References

1. *Chemistry, Starter Investigations (Advanced Higher):* Crystals
2. *Stern, KH: the Liesegang Phenomenon:* Chem. Rev.; 1954; 54(1); 79-99
3. *An Experimental Study of the Liesegang Phenomenon and Crystal Growth in Silica Gels:* A.H. Scharbaugh III and A.H. Scharbaugh Jr. JCE, 1989, 66, 7, 589.
4. *Crystal Growth in Gels:* S.L.Sulb, JCE,1985,62,81
5. *An Interesting Student Project: Investigating Liesegang Rings:* R.A. Schibeci and C. Carlsen, JCE, 1988, 65, 365.

S1/S2 Electronics

There is no perfect set of microelectronics resources for teaching electronics in S1/S2 Science. There are difficulties with whatever approach you choose. However weighing up all of the pros and cons, *MFA* (at least until this new JJM product, the *Angus Systems Board*) continued to stand out head and shoulders above the rest of the pack. Admittedly it is not pupil-proof. But were a kit to be built like a tank, then it would look like a tank rather than a microelectronic system.

With his considerable understanding of teaching electronics and designing electronics kits, Alex Munro (the owner of JJM Electronics and a principal teacher of physics) has devised a new kit – the *Angus Systems Board* - that closely resembles the MFA *Decisions Board*, but which has some modifications and additional features. The chief improvement is to let pupils use the kit with external devices. For instance, a closed-loop control system could be set up causing a train to run to and fro on a track. Thus whereas MFA mainly models abstract systems, the Angus Board, to a greater extent, models practical ones.

Kits	Pros	Cons
MFA and JJM Angus Board	They look the part and are well designed. Cover many basic concepts of electronics. Suitably simple for beginners, yet have sufficient depth to challenge all abilities. Place the emphasis on processes and problem solving, as opposed to learning lots of facts. Can stand on their own. Do not depend on pre-knowledge of electricity or other physics. Excellent self-learning support materials. Can be taught by non-experts who have little understanding of either electricity or electronics. Include facilities to operate external devices (e.g. a buggy). Reasonably robust and reliable. Good guidance available on repairs and spares.	Susceptible to malicious damage. Skilled electronics technician needed to repair faults or damage.

The Angus Systems Board costs £49.85 alone, or an extra £8.95 with its remote sensor pack.

Note that the scope of electronics systems that can be designed with MFA is considerably greater than the ones with the Angus Board.

The latter includes no more than logic, whereas the former, with a set of three boards, also includes counting, display and memory. Thus the Angus Board would be an adequate resource for a short course of about two or four lessons on electronics at S1 or S2. For a course of greater length or depth, MFA would be a better buy.