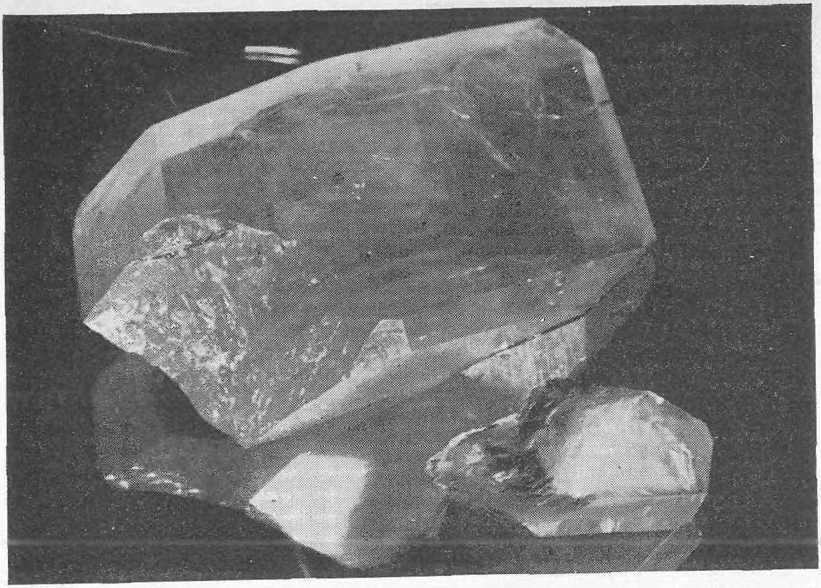
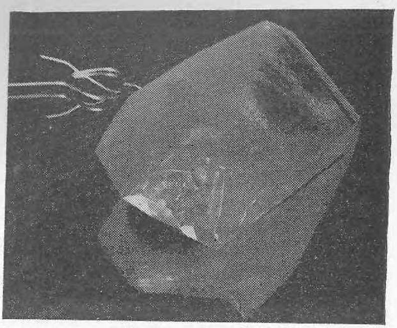


Copper sulfate crystal, deliberately broken, with chip beside it. Photographed on a black ceramic tile.

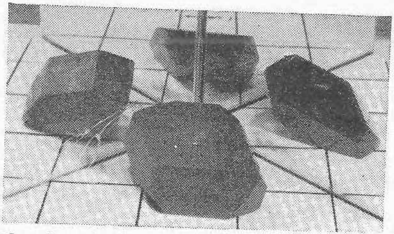
THE SELF-REPAIRING ROBOT

By JOHN W. CAMPBELL

The self-repairing robot must fall somewhere halfway between a simple inorganic system, and a true living organism. The more you study what crystals do . . . the more difficult it becomes to say "Here Life Begins!"



Copper sulfate crystal after three days of healing in the saturated $CuSO_4$ solution. Note that crystalline facets have started to fill in the wound.



The healed copper sulfate crystal . . . and the chip. The chip, too, was put in the growth solution, and healed to a complete crystal.

SOONER or later, we'll have to perfect a system for producing self-repairing machines; surely the self-repairing robot is a standard ideal in science fiction. Perhaps the first step is to define just what it is we mean by a self-repairing entity, to get some idea of just what that goal we're striving for actually means.

Of course, not all robots are the conventional tin-man type; probably the simplest of all present household robots is the little contraption that lives just inside cabinet doors. It reaches out about half an inch, takes hold of the cabinet door, pulls it shut and then holds it there. It's essentially a static machine—just a permanent magnet—but it does perform a dynamic function.

Most of the advances being made today in the direction of real, high-order robots—the self-directing thinking machine type—are being made in the area of solid state physics—in an area where the working units are not man-shaped parts such as gears, shafts and wires, but atoms and molecules. The function of intelligence seems to be very intimately, and probably inherently, associated with complexity—the interaction of billions of decision-making units. If robot intelligence is ever to be achieved, it will involve some practicable method of producing billions of decision-making units at low cost, in minute volume, with high speed, and immense reliability.

Reliability is, actually, the overwhelmingly critical problem. If we had a system that produced only ten per cent functional units, and ninety per cent duds, it would still be fine . . . if those ten per cent functional units could be absolutely guaranteed to *keep on* working. It would mean that a simple test now would make certain that the unit would be functioning at all later times; on that basis you can steadily accumulate functioning units, and nothing but functional units. The great problem in reliability in a complex system is this: if a given component has a reliability such that it will show only one failure in one million operations, then if one million of those units are coupled together, there is only a fifty-fifty chance that the system will work. If ten billion are coupled—and there are ten billion neurones in a human brain—there would be only one chance in ten million that it would work at all!

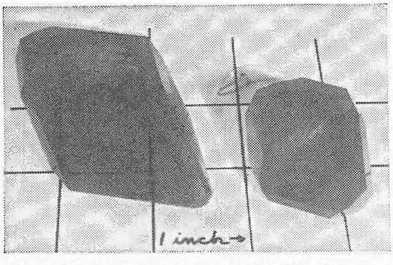
No device whose components are at the gross mechanical level can achieve the required level of reliability; soldered joints don't approach that level, for instance, so that no wired computer could achieve intelligence. Certainly no electromechanical relay approaches the thousand billion operations level of reliability essential for an intelligence-system structural unit.

Currently, work is being done in growing complete electronic circuits as part of modified single crystals. The units are not manufactured; they're grown. The operational parts of the units are at the submolecular level, where the concepts of friction, wear, abrasion, et cetera, have no meaning.*

These crystals are, of course, the silicon and germanium crystals that first came under intense investigation with the development at Bell Laboratories, of the transistor.

The self-repairing robot isn't with us yet, by a long way, at the man-made level. But there are plenty of self-repairing robots already available for home-grown studies—and the term home-grown is literally exact. They cannot be *built* at home, but they can, very easily, be *grown* at home. Quite ordinary crystals, such as copper sulfate, alum, potassium dichromate . . . these are true, self-repairing, and self-constructing robots.

The essential requirements before a robot can be self-repairing can be laid down, even if we can't state how those requirements can be fulfilled. To be self-repairing, an entity must (1) have some sort of awareness . . . understanding . . . memory



The chip photographed in the intersection of two mirrors at right angles, to show all sides. A white ceramic tile, ruled with India ink and sprayed with a lacquer is a highly useful tool in this work.

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*Credit where credit is due department: Robert A. Heinlein, in his story "Universe" in the May 1941 Astounding Science Fiction, referred to "submolar mechanics" as the technique used in producing the control systems of the spaceship that continued to function for over four thousand years. He specifically made the point that submolar systems would have inherent in them the requisite level of reliability.

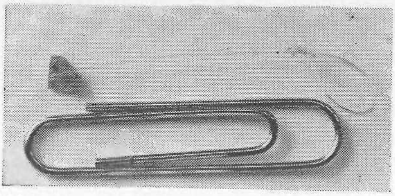
. . . conception . . . call it what you will . . . of What Should Be; and (2) a simultaneous awareness, or what have you, of What Is; and (3) be able to do something to make What Is change to align with What Should Be.

The terms are, obviously, anthropomorphic—subjective. Highly objectionable in any scientific discussion.

Too bad, too . . . they happen to fit perfectly. Call What Should Be the Ideal, the Pattern, the Original Instruction—it still comes out having the meaning What Should Be, because the self-repairing robot must not only have a pattern, but be driven to achieve the pattern—motivated, in essence. The essence of the self-repairing function is to change the improper state to the intended state; both terms inevitably imply a bias, a choice and a motivation.

An ordinary machine has no tendency toward self-repair, because any condition it is in, is, so far as it's concerned, the appropriate and accepted state of affairs. To be self-repairing, the entity must, in essence, have a sense of discontent which is stimulated whenever What Is does not match the Ideal. And that, of course, requires that there be an Ideal of which the entity is aware in some fashion. A living animal heals a wound because the wound is not What Should Be; the organism is stimulated to repair it.

The self-repairing robot must have a similar sense of discontent with disrepair.



Nickel sulfate hexahydrate — $NiSO_4.6\ H_2O$ — seed crystal cemented with polystyrene cement to a piece of eight-pound test monofilament nylon fishline.

Now such ordinary crystals as I mentioned show precisely such characteristics. Some ten months ago, I started studying some of the things crystals will do, strictly on a home-experiment level; the more I've worked with those strictly inorganic crystals, the less I am able to give any objective statement of the difference between living and nonliving entities. None of the usual statements that we get in high-school biology really work —and the more I study crystals, and study what biochemists are doing, the closer the two subjects seem to approach each other.

Usually, it's said that nonliving things cannot grow, reproduce, or heal themselves. Crystals grow, of course, but, says the old biology definition of "living," they can grow only in a medium of their own kind, without transforming that medium into their substance.

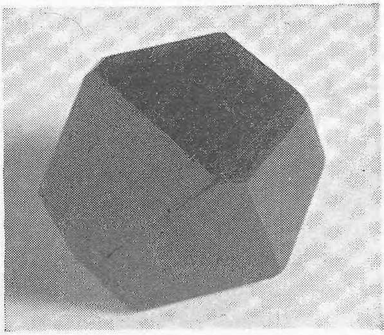
Well . . . a plant, say a yeast cell, can't grow in a medium that contains no carbon. And it can't grow in a medium that does not supply it with chemical energy to drive its biochemical processes. A green plant, which can trap solar energy, still needs an environment which already contains energy to operate its process—in this case, light energy is part of its environment.

But if you add an iodide solution to cupric sulfate, or any soluble cupric salt, crystals of cup*rous* iodide form. The cupric ions have been altered by the forces of the crystallization process to yield cuprous ions, on which the crystals feed.

Take some aluminum sulfate crystals, and some potassium sulfate crystals. Put them in water. In the course of a few days, all of this "food" will have been "digested" and transformed into a new material—alum.

Because crystals are self-repairing, crystals can readily be reproduced by simple fission, just as primitive organisms can. Cut a hydra in half, and you can get it to grow into two hydra. In the photographs on pages 83 and 84 you see reproduction-by-fission in the case of a copper sulfate crystal; the crystal was broken, and the crystalline forces repaired the large crystal. The crystal in the junction of the two 90° mirrors is the chip from the large crystal, which repaired itself, and grew into a daughter crystal.

The seed grown up — six days later. The scale of this shot is about two-thirds that of the preceding — nickel sulfate grows easily and rapidly.



Of course, crystals normally reproduce by seeding. It's been held that living things can start only from living things, while merely allowing a solution to evaporate spontaneously generates crystals.

There's plenty of reason to question that one.

Some years ago, physical chemists at an American university wanted to study solid glycerine. Now the handbooks say that glycerine freezes at about room temperature—68° F.—but that means pure, water-free glycerine. Since glycerine is rather frantically hygro scopic, water-free glycerine is not a normal occurrence, and the water naturally lowers the freezing point of the solution.

The chemists purified their glycerine, dehydrated it thoroughly, and chilled it. It didn't freeze; it just got viscous. They jolted it, stirred it, and did all the things crystallographers do in inducing reluctant substances to crystallize. They cooled it in liquid air; it turned to a glassy, but still amorphous, solid.

Finally they wrote to the German university where the original published material on glycerine, solid state, had been written. What was the trick of freezing the darned stuff?

The German replied that he could not understand their difficulty; one simply chilled it, and it froze. Perhaps the Americans didn't have pure glycerine? Herr Doktor was sending a sample along. . . .

And since that sample arrived and was opened, nobody has been able to keep glycerine from freezing in that laboratory. It no longer goes into a glassy state even if they want it to.

Crystals, apparently, have seeds—microscopic spores that are far more subtly submicroscopic than the smallest virus particles. And without them, the crystals don't form any more spontaneously than do living cells!

Again: For several years, the researchers at the Bell Laboratories worked on the problem of growing piezoelectric crystals for use as tuning elements in electronic circuits. Quartz crystals are just about ideal . . . but the natural supply was running out, and nobody knew how to grow quartz crystals. Over a period of a couple of years, the Bell Labs people worked out techniques for growing perfect, flawless crystals of ethylene diamine tartarate—crystals that did the required job very nicely. Production-line methods were developed, and regular production of the crystals was operating in full swing for several years . . . until one day, the thing came to a grinding halt. Their perfect crystals began coming out with a few small parasitic crystals, at first, then more and more of the parasitic crystals.

Investigation showed that the crystal parasites were a never-before-heard-of ethylene diamine tartarate monohydrate. There had been a mutation, and a new species had appeared in their nutrient broth—saturated ethylene diamine tartarate solution. That was the end of the EDT crystal production.

Fortunately, work on the quartz crystal problem had finally cracked the problem, and Bell Labs was able to produce quartz crystals commercially.

So . . . crystals arise spontaneously, unlike living forms? Anybody able to prove that conclusively? There's lots of evidence that they *don't* arise spontaneously; that there must be pre-existent seeds! The Bell Labs people had worked with tens of thousands of gallons of solution, with thousands of pounds of crystals . . . and done so for several years. Then suddenly, the new crystalline form appeared, and that was the end of the system.

The most important characteristic of a crystal is its form, its pattern. Alum is one of the easiest to grow; anyone starting home-grown experiments should start with that. Alums alone offer enough different problems and challenges to keep you going for years.

An alum is, essentially, a double sulfate of two metallic ions; one is a univalent ion such as potassium, sodium, ammonium, caesium, et cetera, and the other a trivalent ion such as ferric iron, chromium, or aluminum—the one which gave its name to the family. You might write the formula of an alum as:

$$\left.\begin{matrix} K \\ Na \\ NH_4 \\ Cs \\ \cdot \\ \cdot \end{matrix}\right\} \left.\begin{matrix} Al \\ Cr \\ Fe \\ \cdot \\ \cdot \\ \cdot \end{matrix}\right\} (SO_4)_2 : 12\ H_2O$$

The dots can stand for a variety of odd and esoteric elements such as thallium and iridium. Now the alums *do not exist in solution*; the solution is simply potassium sulfate and aluminum sulfate—the potassium and aluminum ions are in no way coupled in solution. It's the crystallizing forces that link them in the special alum arrangement.

Aluminum-potassium alum *always* contains exactly one Al atom for each K atom; the proportions are invariant.

But any two alums can be mixed in any proportions, and will co-crystallize together into single, perfect octahedral crystals of alums, in any proportions. Alum crystals form double square pyramids, base to base; $KCr(SO_4)_2 \cdot 12\ H_2O$ is deep purple . . . in sand-grain size crystals. In full-sized crystals the color is so intense, they're glossy, jet-black, and very handsome. Potassium-aluminum alum, $KAl(SO_4)_2 \cdot 12\ H_2O$ is glassy-clear and colorless; the crystalline form is the same as that of the diamond crystal. Various other alums have varying colors, depending on the trivalent element used. Since iridium sells for several hundred dollars an ounce, I don't recommend working with the rather soluble iridium alums, but the others make some interesting possibilities.

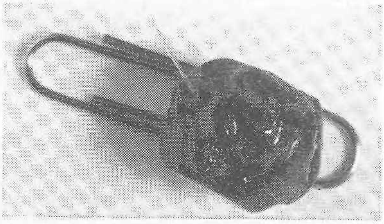
Chrome alum is so deeply colored, the crystal looks black; it can be co-crystallized in any proportions with aluminum alum, to yield crystals ranging from deep purple through pale bluish to water-white. A mixture of five parts Al alum to one part Cr alum gives a deeply colored green solution; remember that alums do not exist as such in solution—and chromium sulfate, unlike chromium alum, is green. But the crystals formed from that mixture are a beautiful amethyst.

At this point, some remarkable crystal-growing tricks become possible. If you make up a solution of the Al alum, one of straight Cr alum, and one of the mixed Al-Cr solution, you can start with a seed crystal in the Al solution, move it, after it's grown a bit, to the Cr alum solution, and let it grow some more. After a thin layer of the deep purple Cr alum has been formed, move it back to the Al alum. A layer of glassy clear Al alum forms. Move it back to the Cr alum for another thin layer of the deep purple. A little experimenting, and you can grow startlingly beautiful zebra-striped crystals—with the stripes actually layers of purple and clear. The whole can be finished off in the mixed alum, for a crystal-clear coating of amethyst.

Incidentally, the Cr alum is not stable in air of ordinary humidity; it will stand up nicely in an East-coast summer, with its usual sticky humidity, but won't last through the winter. Aluminum alum, however, holds onto its water of crystallization much more firmly. If you expose the Cr alum to air, it turns into a lavender powder and crumbles away. The five-to-one mixture of Al-Cr alums has the resistance of the Al alum, and is stable in normal climates.

The neatest way to show, visually, the repair power of crystals makes another use of the mixed-alums technique. Grow a crystal of either the amethyst mixture, or of Cr alum to about a one-half inch size. Then grind off one corner (it won't cut—the stuff is too brittle—but you can grind it down on sandpaper) until it is quite clearly a broken, or wounded, crystal. Now put it back in the clear alum solution; growth will start and the crystal-repair faculty begins operation. In a matter of a few days, a thin layer of water-white crystal will coat the whole crystal, while a mass of white "scar-tissue" forms over the broken corner. In another few days, a thick layer of glassy crystal encloses the whole structure—including the white scar-tissue area.

Cobalt sulfate—shown here—and a number of other substances I haven't been able to grow in clean, single crystals. The cobalt sulfate insists on forming lumpy, polycrystalline masses.



Unfortunately, I'm stuck with the fact that this is a highly visible, but practically unphotographable gimmick; it can be done with a truncated amethyst crystal in plain alum repair solution, and photographed in color very nicely. Trouble is . . . Analog can't as yet afford inside color plates. In black-and-white, the jet-black internal crystal looks like a simple hole, a break, in the crystal, and is about as uninspiring as a hole in anything else.

A better way of showing the process of crystal repair photographically is that series of the copper sulfate crystal on pages 83 and 84. The large crystal was broken; the chip is shown beside it. This series can't be done visually, of course; it's something like the fabled French museum that had on display the skull of Napoleon as a small boy. You can't both repair the crystal and have it on display as a broken crystal.

Many crystals cleave very nicely—nickel sulfate hexahydrate, for example, can be cleaved into neat, flat plates. This doesn't make much of a display as a "broken crystal"; copper sulfate, however, has an extremely complex crystalline form, and doesn't cleave. The fracture surface really *looks* like a fracture.

When put back in the "nutrient medium," healing starts—and the healing mechanism is quite clearly visible. Copper sulfate grows in beautifully transparent, deep-sky-blue crystals—which makes it more than somewhat difficult to photograph, incidentally. The repair process starts by throwing out new facets and lines and edges—sharply, regularly geometric pieces grow over the irregular fracture surface. Gradually the pieces grow together; fewer and fewer geometric-angular planes and faces remain, and finally the whole zone has been filled in with a solid mass of copper sulfate crystal, and has grown into perfect alignment with the undamaged part of the crystal.

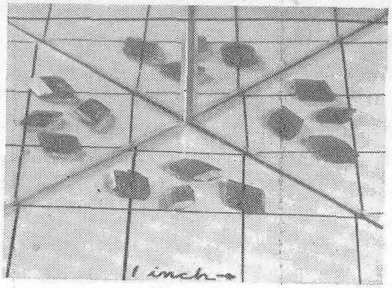
But . . . it leaves "scar tissue." Whenever a crystal's growth is stopped and restarted, there's almost sure to be a veiling—a slight clouding, under the best of circumstances. (That's what makes getting perfect, flawless crystals so difficult—the growth must be continuous, or you get "growth rings" throughout the mass.) When the crystal heals, visual examination will show an area of veiling where the "scar tissue" repair mechanism grew together.

The last picture of the $CuSO_4$ crystal series shows the broken-off chip also "repaired" into a full-size crystal, in the juncture of two mirrors at 90°. The shape of a copper sulfate crystal is too complex to describe; only this four-sided view of it gives a visual suggestion of how intricately the facets and edges interact. On page 92 is a similar shot of a group of well-started copper sulfate seed crystals; note that each has almost, but not quite exactly, the same shape—but each has exactly the same arrangement of facets.

Properly grown, crystals come out of the solution with edges that are mathematically straight lines, and are razor sharp—handle with care!—because those edges are just about one molecule thick. The best technique I've found for getting them out and retaining their beauty is to wipe them off immediately—within seconds—on soft absorbent paper tissue. If you wash them, the water etches the surface and dulls the final sparkle. If you let them dry, the remaining solution deposits minute crystals that dust the brilliant facets.

Whatever the crystal, however, *form*, rather than *substance*, dominates; that's one reason why the alums are so interesting to study. The *form* of an alum crystal is the octahedron; the *substance* is relatively unimportant. It's as though a man building a house needed timbers of certain sizes—two-by-fours, eight feet long, say. Whether they were oak, pine, mahogany, or fir would make very little difference to him in getting the shape of structure he wanted, so long as they had the right shape. There would be limits, of course—balsa wouldn't do, because it's too soft and weak. Lignum vitae wouldn't do, because nails can't be driven into it.

Ferric potassium alum can—just barely—be prepared; ferric ammonium alum is stable and crystallizes nicely. Apparently ferric postassium alum is stretching the limits of the material requirements of the alum



Copper sulfate seed crystals.

pattern. But *alum* is not a chemical substance; it's a pattern of crystallization.

In the cloud-seeding experiments, this phenomenon of pattern-vs.-substance was applied. The shape of a silver chloride crystal—the inter-atomic spacings, the pattern—very closely matches the pattern of an ice crystal. It's not the same—but so closely similar that it can act as a matrix on which supercooled water molecules can settle and start forming ice crystals. It's a "stereo-catalyst," in that it has the correct three-dimensional pattern to act as a matrix for the desired crystallization.

And what's an enzyme in a living organism? Currently, it appears to be a stereo-catalyst that has the correct spacings to allow a desired molecule to form.

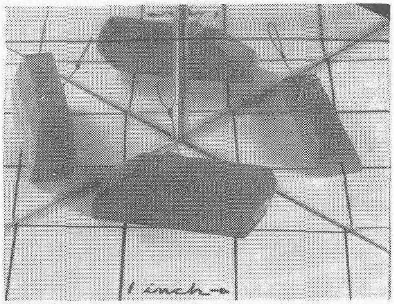
When men first made plutonium nitrate, they were producing a molecule that never existed on Earth before. Yet they were able to crystallize it—and obviously, they didn't have any seeds of that crystal to start with? Why didn't they wind up with the sort of problem the glycerine presented—no seeds: no crystals?

Chemically, uranium and the transuranic elements so far studied, are very closely similar—very much as the whole group of "rare-earth elements" near the middle of the periodic table are. It's probable that uranium nitrate crystal seeds could serve as matrix seeds for plutonium salts. If silver chloride can serve to start ice crystallizing, it's quite probable that something fairly common can serve as the trigger for plutonium and similar synthetic element compounds.

Cases like that of the ethylene diamine tartarate are decidedly unusual—probably because of that phenomenon of stereo-catalyst effects by quite alien substances.

However, the single instance of ethylene diamine tartarate demonstrates that there may well exist dozens of *possible* crystalline forms that no one has ever observed, because they've never happened—because the necessary "mutation" has never occurred.

Potassium bichromate — a brilliant flame-orange color.

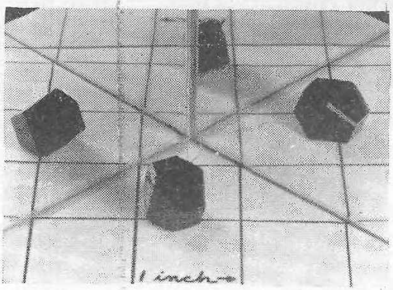


Important as form is, crystals are, like living things, able to adapt to environmental stresses. One of the simplest adaptations is shown by—again! alums. In neutral, or slightly acid solutions, they form octahedra; in strongly acid solutions, however, they crystallize in an entirely different shape, producing dodecahedra.

If you are not a practicing advanced inorganic chemist, you're apt to learn that inorganic chemistry, as taught in high schools and college courses other than advanced inorganic, neatly lead you all around the really tricky part of the subject. The most interesting crystals all involve the transition metals—chromium, manganese, iron, cobalt and nickel and copper. These elements are casually mentioned in ordinary inorganic chem textbooks, and then very hastily by-passed. It just happens that practically all the colored inorganic compounds—other than thoroughly insoluble oxides and sulfides—are compounds of those transition elements . . . and their chemistry is not anywhere near as simple as ordinary inorganic chem courses may have led you to believe.

Chromic salts are green; chromates give brilliant yellow, and dichromates are brilliant orange. Potassium dichromate crystals are beautiful, flaming orange.

Manganese sulfate solution is a sparkling-clear solution of delicate flesh-pink color. Manganates are green, and the permanganates are intensely magenta-colored. (Chlorates and perchlorates by contrast are perfectly colorless—though perchlorates and permanganates are, like the alums, isomorphous and can be co-crystallized. Thus potassium permanganate, intensely magenta, and potassium perchlorate co-crystallize in all porportions, to give crystals ranging from colorless through pinks to deep violet.)



Nickel sulfate hexahydrate—emerald green.

Iron salts tend toward the greenish—ferrous—or reddish-brown—ferric. Nickel salts are mostly emerald green, while cobalt salts run to reds, lavender, and blue. Cobalt sulfate is garnet-red, with brown undertones; the basic carbonate is a really remarkable lavender.

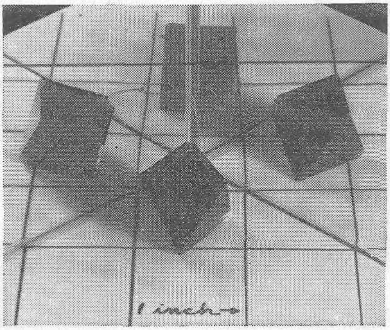
Copper, of course, gives blue and blue-green colors. The sulfate is a magnificent blue jewel; it's a shame it isn't hard and water-proof! But . . . it says in the chemistry texts that copper-lithium chloride makes deep ruby-red crystals. Mine came out dirty chartreuse.

Add potassium dichromate to nickel sulfate, and the greenish-yellow crystals that separate will be, obviously, nickel chromate. Only . . . they happen to be nickel-potassium sulfate, and nickel chromate won't crystallize. (Nickel has two valence possibilities, and in the presence of the powerfully oxidizing chromate radicle, it apparently can't decide what to be, and so never does get around to crystallizing decently.) Nickel-potassium sulfate is much less soluble than nickel sulfate, so that forms and crystallizes out.

Alum is, as I say, the place to start . . . but the brilliantly and beautifully colorful salts of chromium, manganese, nickel, and cobalt are attractive. Copper sulfate crystallizes well and easily; just make sure you use distilled water, and keep contamination out. And add a few cc's of sulfuric acid per litre of solution; copper is highly susceptible to alkaline contamination, and then produces veiled crystals.

Nickel sulfate produces beautiful green crystals quite readily. However . . . here beginneth the tricky stuff. Nickel sulfate forms two hydrates; $NiSO_4 \cdot 7H_2O$ and $NiSO_4 \cdot 6H_2O$ The heptahydrate forms long, nearly-square cross-section bars; at room temperature, the heptahydrate is less soluble than the hexahydrate, and so it crystallizes out rather than the hexahydrate.

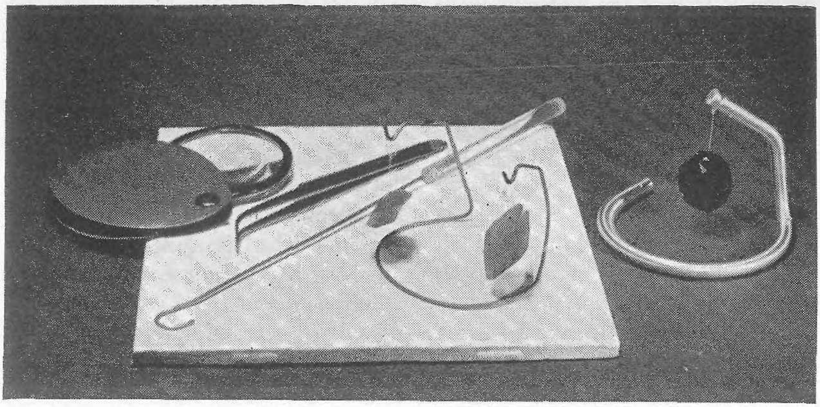
Mixed (5 to 1) potassium-aluminum and potassium-chromium alum crystal. The crystal is a true amethyst color, and grows easily.



However, the heptahydrate is unstable in room air, loses water of crystallization and crumbles to a greenish white mess. Also, the crystal bars refuse to form up the ends of the bars—they are always ragged and unfinished.

Above 90°F. however, the hexahydrate is less soluble than the heptahydrate, so the hexahydrate tends to crystallize out. If a heptahydrate crystal is put into a saturated solution of $NiSO_4$ at 100°, it will slowly dissolve, while a heptahydrate crystal grows at its expense. The hexahydrate is a slightly different color, and forms the squared-off-barrel shaped crystal shown in the two-mirror shot on page 93. It forms up very nicely, and the crystal is stable in air.

There is one temperature—about 85°F—at which the two forms are equally soluble; at that temperature, the hexahydrate and the heptahydrate can be grown simultaneously in the same solution. Makes an interesting stunt—two very different crystals growing from the same solution under the same conditions. (Hm-m-m . . . Hey, Aristotle, what's this about nothing both *being* and *not being* at the same time in the same respect . . .?)



Tools of the trade. A white and a black ceramic tile make excellent working surfaces. Hand lens and tweezers are essential; the "fishing hook" is, like the smaller double-ended crystal support, made of tungsten wire, about #15 gauge. Tungsten and platinum are about the only metals that are immune to all the solutions you'll be likely to work with. The single-ended "cobra" is made of one-quarter inch Carpenter #20 Stainless; no ordinary *"18-8 stainless steel" will resist nickel or manganese* sulfate solutions—and *no* stainless will resist copper chloride.

Most of the tools required are quite simple and obvious—convenient glass jars, largely. Pyrex beakers, of course, are made for that sort of thing, but aren't necessary. Peanut butter jars work fine. And I recommend pieces cut from rubber-floor tile as lids, if you use the slow-evaporation method of growing crystals. You can cut slots of varying size in the tile-pieces, and regulate evaporation quite conveniently.

Filtering is going to be necessary; your local photography shop may prove a convenient source of a number of chemicals and items like polyethylene filter funnels. (Chrome alum is a standard stop-bath in photographic developing.) Chemex coffee filters are more widely available than standard chemical filter paper, and are perfectly good filter papers, of course.

Preparing solutions is not quite the simple matter of stirring chemicals into distilled water that it at first seems. (And use distilled water; it'll save you trouble and expense in the long run. Tap water has God-knows-what contaminants in it—usually alkalies that happily precipitate copper, nickel, and chromium, or chlorides, perhaps a trace of free chlorine to oxidize solutions. In some areas, you'll find that a little sulfide goes a long way to ruin things.)

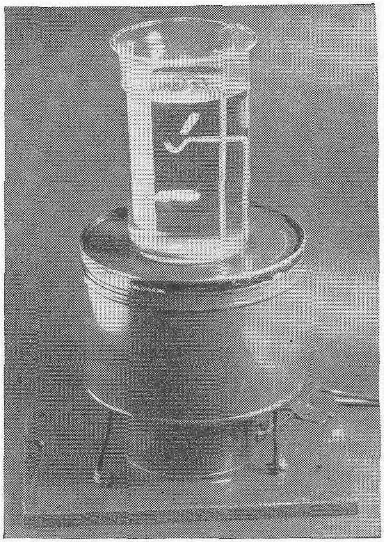
The simplest way to prepare your

solution is to heat the water to about 150° or so—you don't need to take it up to boiling—and stir in your crystals. When the solution won't dissolve any more, pour it off from the crystals into a clean container, with just a few small crystals in it.

Now wait three days.

On that I'm not kidding. I don't know why; I'm simply stating experience. For some reason, it seems to take approximately three days for a solution to stabilize. If you put a seed-crystal into a freshly prepared solution, it may dissolve for no imaginable reason, or it may suddenly start sprouting dozens of unwanted side-crystals, while the solution suddenly starts snowing tiny crystals.

Half a cherry can, some heavy-gauge copper wire, and a coffee can make a highly useful stove for preparing solutions. A ceramic socket allows use of anything from 8 to 100 watts; highly useful for cooking up solutions, and rapid evaporation of too-dilute solutions.



Prepare a stock-reserve of the solution, to make up losses as the solution evaporates and deposits crystal material. Keep the reserve in a vapor-tight bottle standing near the working solution, and let it age, too. If you try adding a freshly prepared solution to your working solution, you can expect trouble. You may not have any . . . but you can't count on luck. If you are working with a warm solution—that nickel sulfate, for instance, running at 100° or so—then the reserve solution must be kept at the same temperature—and kept there for about three days, too.

Copper sulfate and alum are pretty rugged solutions; they work nicely under not-too-good conditions. The same can not be said for nickel sulfate, and is definitely not true of potassium dichromate. I have not yet been able to get a decent crystal of bright-yellow potassium chromate—and not for want of trying. Cobalt sulfate still defeats me, too; it insists on making large, knobby polycrystalline masses, instead of decent monocrystalline units.

Potassium dichromate will crystallize into large, brilliant crystals *if* (1) the temperature is held fairly close to constant—within about 2°F, and (2) it is allowed to crystallize only very slowly. Keep the rate of evaporation low, and constant.

That can be done during the winter

in a thermostatically controlled heated house. In summer, it calls for a thermostatically controlled air-conditioner, plus a dehumidifier. To grow a crystal of $K_2Cr_2O_7$ an inch and a half long takes about two months. An alum crystal of equal mass can be grown in five days, if you push it a little.

And with the dichromate, you *must* use an old solution; let it age a week or so before trying anything really serious with it.

Getting seeds for the solutions is fairly simple, but tools are needed—and you have to invent and manufacture most of them. Corrosion is going to be your major enemy—and not always the solutions you expect to be corrosive. Potassium dichromate, for instance, is an extremely powerful oxidizing agent—it'll cause ulcers if you let it stand on your skin any length of time, and the dust of spilled-and-dried $K_2Cr_2O_7$ is decidedly bad for eyes and lungs—wipe it up carefully. Yet that solution isn't at all hard to handle, so far as corrosion goes.

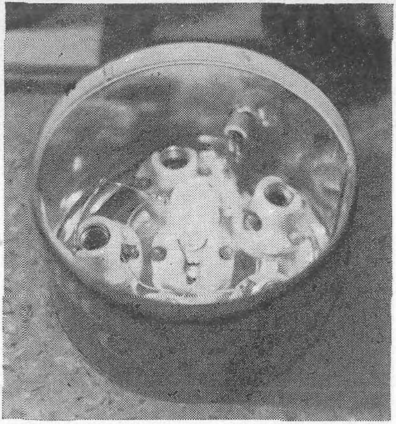
The one that really chewed holes in things was the mild little nickel sulfate! $NiSO_4$ is really quite soluble; it takes about two pounds of $NiSO_4 \cdot 6H_2O$—the commercial form in which nickel sulfate is sold—to saturate one litre of water. The resulting solution is dense, and highly concentrated. You need to keep it slightly acid, because of the insolubility of the nickel hydroxides and basic salts—and that means that it is, effectively, a very concentrated solution of sulphuric acid, so far as corrosion of metals goes. It chews holes in most types of stainless steel. Nichrome wire—used in electric heating resistances—is a type of super-stainless steel; it goes to pieces in an hour or so in nickel sulfate. Lead dissolves, and makes the solution cloudy with white lead sulfate.

Manganese sulfate also proved to be highly corrosive to metals.

Copper sulfate can be handled very readily; copper wire and tools may corrode, but not seriously, and the corrosion will be nothing but a little more copper sulfate, so who cares.

Yet these metal-corroding solutions aren't as corrosive to organic matter as the dichromate solution! The dichromate, because it is a powerful oxidizer, immediately forms a coating of oxide on metals exposed to it . . . and the coating stops further action right there.

Plastics are your best materials, within their limitations. Since most of the standard plastics are hydrocarbon-based, they like wax, simply don't actually get wetted, and no corrosion results. Your local radio-TV shop probably has the "Radio Master" catalogue; in that, you'll find listed polystyrene rods, sheets and tubes; they're used in electronics because of the high insulating abilities of polystyrene, and they're reasonably cheap, and easy to work. Polystyrene cement will truly weld two pieces of poly together—and polystyrene cement proved to be the best glue for



One coffee can plus six small ceramic sockets and a supply of Christmas tree bulbs make a low-energy stove for maintaining solution temperatures.

sticking seeds crystals on nylon support strings.

If you can work glass, that's even better, for most things, than plastic—but plastics can be worked with hand tools readily. The major trouble with plastic is that in dense solutions like *nickel sulfate, the darned stuff floats,* and you need an anchor weight to haul it down. Since not even lead is immune to the attack of some of the solutions, this gets to be difficult; ingenuity is a marked advantage in this hobby! Paints and lacquers over metal usually prove to have pinholes, with resultant peeling. I used a piece of three quarter inch poly rod, drilled with a half-inch hole, poured full of solder, and polystyrene-cemented to a polstyrene plate for my most-resistant.

You need some kind of plate to grow seed crystals—a plate you can *lower into the solution,* and *haul* out for selection of crystals. Rig it your own way!

You'll need needle-nose tweezers—preferably high-quality stainless-steel—and a magnifying glass, to pick out the best of the minute seeds; they'll start forming "spontaneously" from your solution after it's had a chance to age for a few days, when evaporation starts concentrating it. Keep the best seeds, and return them to the working solution. Pick 'em over again about twelve hours later. It takes only about two or three days to get a satisfactory supply of seed crystals.

Put all but one or two in a small bottle, and then start work on your growth project. Item The First is gluing the seed to a support thread. I use eight-pound-test monofilament nylon fishline. It won't corrode, and is so smooth it discourages unwanted volunteer crystals. It's also slippery as an eel, and as springy as a good *spring steel, and will drive you to* new cuss words before you get a technique for tying a loop in it where you need it. And what you want is a loop to suspend it at one end, and a knot at the other end, about an inch away. The knot is then smeared with a small drop of polystyrene cement, and that, in turn, stuck onto your seed crystal.

The resulting attachment is weak—but your crystal is small; when it grows, it grows around the string, and anchors itself very solidly to it.

The crystals must, of course, be dry when you apply the glue; they can be dried by rolling them gently between your fingers in a piece of paper tissue of the *Kleenex* variety, and then air-drying for a few minutes. *If* your solution is truly saturated, when you put the crystal-and-string into it, the crystal will be insoluble, so the glue won't come loose. If the solution isn't completely saturated, you'll find that out in about five minutes; the glue will peel off and drop the crystal. If things are as they should be, the crystal will grow happily and firmly onto the cord.

Incidentally, I tried various other cements, glues, and stickums; there may well be others that work, but polystyrene cement did better than anything else I tried—to and including the fabulous epoxy resin glues, which peeled off with frustrating regularity. This despite the fact that polystyrene cement is, generally speaking, a very poor sort of glue; it's perfect for polystyrene, and nothing else except crystal growing!

Now cometh the stinker: the matter of what to hang the nylon string and crystal from. Again—if you can work glass, a "cobra" made of glass rod will work beautifully in any solution, having only the difficulty of being brittle. Otherwise, you can use heavy-gauge aluminum wire in the alum solutions—it may corrode, but it just makes more alum. Copper wire in copper sulphate, chloride, or what-ever. But you don't get nickel wire very readily, and such things as manganese and chromium wire are definitely not to be had. Of course, platinum wire would be just fine . . . at about five dollars an inch. I got hold of some Carpenter #20 stainless steel; this is a super-resistant, ultra-corrosion-proof stainless, and it will stand up to nickel and manganese sulfates . . . but in a cooper chloride solution it had corrosion pits in about thirty seconds. If you can get titanium wire, or zirconium wire, they'd do nicely.

I found that Central Scientific Company (Cenco) stocks tantalum and tungsten wire, and at not-impossible cost. Tantalum is malleable, somewhat soft, and proof against any ordinary acid medium; tungsten will resist anything short of boiling, concentrated potassium hydroxide. No acid will attack it, and nothing but the most virulent alkalies. It costs about five dollars for four feet of approximately #18 gauge, which is stiff, strong, and entirely satisfactory.

The one difficulty is that tungsten, at room temperature, is about like steel at liquid hydrogen temperatures; it's brittle. The wire can not be shaped at room temperature—it shatters. It can, however, be bent as readily as any ordinary metal if heated red hot; an ordinary alcohol lamp will do the job, and two pairs of pliers—with due caution not to have a red-hot length of tungsten wire whip around at you—does the job readily. The tungsten oxidizes somewhat in air at red heat, so try to keep the red-heat time down, but the oxide won't cause any trouble. It clings very solidly, and is as corrosion-resistant as the metal; it, too, yields only to boiling, concentrated KOH.

The tungsten wire can be shaped into wire "cobras" with either one, or two hooks for supporting crystals; it also makes a highly useful fishing-hook tool. Take a six inch length of the wire, form a hook in one end, and heat the other end and thrust it into a four to five inch length of quarter-inch plastic rod an inch or so. When the rod cools, it's welded in place solidly; the resultant tool is useful for fishing out the wire cobras, slipped-off crystals, and similar emergencies.

For heating solutions to mild temperatures—maintaining nickel sulfate at 100° for instance—or evaporating off excess water, a low-wattage electric stove is very useful. You can buy one for about seventy-five dollars; mine consists of a coffee can in which I mounted six porcelain miniature sockets. By screwing in from one to six Christmas tree bulbs, I have six levels of heating available. The top of the can is sprayed flat black inside and out, while the can itself is left with shiny metal sides; practically all the heat output of the bulbs appears in the top, where you want it.

Another tool of the greatest importance is the "Chemical Rubber Handbook;" it's got a wonderful list of inorganic compounds, their solubilities, crystalline forms, etcetera. Before starting on some interesting compound, look it up—some of 'em have incredibly perverse behavior. Manganese sulfate, for instance; it forms a beautiful flesh-pink solution, and crystallizes rapidly and smoothly into sparkling-clear rhombic crystals of the same delicate pink. They are gorgeous crystalline jewels . . . so long as you leave them in the solution. The stuff is efflorescent in air; it loses water of crystallization rapidly, and turns to an off-white powder. I tried enclosing the crystal in four coats of spray lacquer; it lasted three days before the creeping crud of efflorescence ruined it. There are special casting plastics, intended for mounting biological specimens; unfortunately they call for curing at about 130°C.—and $MnSO_4$ has remarkable characteristics. There are about half a dozen hydrates; one form is stable only between about —10° and O°C.; the one stable at room temperature is unstable above about 40°C.

If you have some excess $MnSO_4$ crystals in a nice, clear solution and want to get them into solution by warming the solution to about 150°F. —what happens is that the whole solution turns sort of milky-pink, it snows off-white microcrystals, and most of the stuff comes out of solution. At the higher temperature, a different hydrate forms, and it's not as soluble, so down it clanks. So now you cool off the solution again, and add a little water, and stir. And this means stir for about twelve to eighteen hours, with an electric stirrer, before you get the nice clear, sparkling solution again.

The pink solution, incidentally, is viscous—it has an oily thickness effect. Pour water into it, and the water floats on top; it'll continue floating there for days, if you don't stir it.

Then there are other crystals that would be nice . . . if they weren't deliquescent. i.e., they soak up water from the air, dissolve in it, and turn into a puddle.

I'm still trying to find an inorganic crystal that is a true, bright red, and is neither violently toxic, some platinum, gold, or iridium compound at fifty dollars a gram, under AEC license provisions, highly explosive, nor deliquescent. Ferric thiocyanate might do, if it weren't deliquescent. The textbooks say that copper-lithium chloride forms ruby-red crystals; mine formed dirty-chartreuse crystals. (And rapidly ate holes in my stainless steel stirring rod.)

Any advanced inorganic chemists around who can suggest a practical true-red inorganic crystal? Or, for that matter, a practical pure-yellow crystal? Potassium chromate is true yellow—but it is not at all co-operative when it comes to crystallizing. Cobalt gives a dirty red—not a sparkling jewellike red.

A few final suggestions:

When I started this experiment, there was no satisfactory how-to-do-it book available. In January, 1960, however, "Crystals and Crystal Growing," by Alan Holden and Phyllis Singer was published by Doubleday Anchor in a paperback edition, selling for $1.45. It contains a lot of directly useful data on methods of crystal growing, and is strongly recommended.

The photographs for this article were taken with either a Mamayaflex Professional C 2¼ x 2¼ twin-lens reflex, which allows focusing without attachments to nearly 1-to-1 size, or the Nikon F 35mm camera with extension tubes. I regret that the high cost of color plates makes it impossible to do justice to the brilliant color of the crystals. Almost any single-lens reflex 35mm camera, with either lens-tube extensions if available, or with close-up auxiliary lenses, can get beautiful Kodachromes of the growing crystals.

With many of the crystals, it is not *necessary* to start seeds, and glue them to supporting nylon lines. Hang a weighted nylon thread from your wire crystal support, with a small droplet of dried, transparent glue on the thread as an irregularity. In the course of a few days in the growing solution, a "volunteer" crystal will usually grow of its own accord on the irregularity. Keep excess, unwanted crystals off the thread, snip off the no-longer-needed weight —the thread would tend to float without it or a crystal attached—and let the crystal grow. This is, incidentally, the only way you can get a perfectly flawless crystal; if the seed is grown separately, attached, and then grown, the crystal will have veils in the seed, and at the attachment. This system just takes more patience.

Potassium dichromate produces strikingly beautiful flame-orange crystals; they're anhydrous, and perfectly stable in air. But dichromates are extremely powerful oxidizing agents; paper filters tend to react with hot dichromate destructively. And unfiltered dichromate solution tends to produce veiled crystals. Solution: go to the local tropical-fish store, and buy glass-fiber for filtering. Use rubber work-gloves for handling the stuff—it goes into fingers easily, but getting it out is hell. Cut off—scissors do it neatly—a small piece, stuff it in the neck of a polyethylene plastic funnel, and pour your solution through it twice. First time through, any loose glass fibers, general crud, et cetera, tends to get lodged in the fiberglass plug; the second time through you have a really fine-grained filter as a result.

Some of the materials you may want to use are available in local drug and/or photographic stores, hardware shops, et cetera. Less common chemicals and materials, such as the tungsten wire, you'll have to order from specialized scientific supply companies.

Central Scientific, 237 Sheffield, Mountainside, New Jersey, is an example—and their policy is typical. They *will not sell to individuals,* and the minimum mail order is fifteen dollars. The reason for the minimum order is understandable; the not-to-individuals policy stems from experiences of this type: If someone sends for two pounds of sulfuric acid, two pounds of nitric acid, and a supply of glycerine, it might be he wanted the sulfuric acid for his storage battery, the nitric for cleaning stains off of stainless steel, and the glycerine for a hand lotion . . . but it sounds too much like someone about to try cooking up some home-brew nitroglycerine. And potassium chlorate, sulfur, zinc powder or charcoal sounds like someone with misguided ideas on home-brew rocket fuel. (That mixture is somewhat less safe to handle than nitroglycerine.)

If you have a company, send in your order on letterhead stationery; otherwise your local school department will gladly cooperate on such a project as crystal-growing.

THE END

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