

THE PEDAGOGUE AND INDUSTRIAL CHEMICAL RESEARCH

D. B. KEYES

University of Illinois, Urbana.

Very little has been written on the general subject of how to do chemical research. It is, therefore, no wonder that the modern teacher in our colleges and universities has paid little attention to the subject of teaching methods for chemical research.

The modern scientific pedagogue will explain in infinite detail how such subjects as mathematics, English, and foreign languages should be taught. Every possible reaction of the average student to every possible system of teaching has been noted and recorded. On the other hand, the student taking up research for the first time is turned over to a scientist who, nine chances out of ten, has very little or no interest in pedagogy. One of two things happens: either the student is allowed to flounder helplessly, to sink or swim depending upon his own brains and good fortune, or he is told exactly what to do every day. In the latter case, he becomes a mere automat and no training of any kind is necessary. Loyalty, perseverance, a strong back, and a weak mind are the outstanding desirable characteristics.

Once the student has graduated and gone to work in the research department of a great industry, he will find to his surprise that industrial chemical research is no longer a guessing proposition. He learns that problems are not attacked by hit or miss methods. It has long been supposed that shotgun methods were still prevalent in industry. This is probably not the case. Research labor is too expensive to be used in this old German method. The method itself was extremely simple, for example, to develop a certain process or product a hundred Ph. D. chemists were hired and put on the job to try all of the possible permutations and combinations, hoping that they would find a combination which would be satisfactory in a finite length of time.

It is the purpose of this article to show some of the popular methods of research now in use in our great industrial chemical laboratories.

Probably the most common type of industrial chemical research is the invention of a new and better process to produce an old and well-known product. The author has analyzed a great many developments of this type, and without exception they have an underlying fundamental method of attack. The strange part of it is that this fundamental method of attack proved to be identical in all the cases examined.

An outline of this method follows:

1. A search of the literature.
2. The selection of a possible process, whether feasible or not, which has the best economic foundation for the particular conditions that have to be met.
3. A list of all of the apparent factors. (By factors are meant pressure, temperature, concentration, and the physical and chemical characteristics of the various substances involved in the reaction.)
4. A list of all the apparent variations of these factors.
5. A selection of the most important factor from the first list, recognizing the basic requirements of this particular reaction.
6. A selection of the best means of varying this factor.
7. The actual experimentation. (If unsuccessful, another selection should be made of the means of varying the factor or if preferred, another factor should be selected to be varied.)

This outline can be best explained by going over one or two examples. Years ago there did not exist a satisfactory process for the cracking of petroleum in the production of gasoline. It was known at that time that petroleum could be heated in an ordinary cylindrical still under pressure and gasoline would be produced. Unfortunately, in every case a side reaction produced large quantities of coke which were deposited on the bottom of the still. This deposit produced poor heat transfer at that particular point, resulting in a hot spot. In other words, the metal at this point facing the fire would be considerably higher temperature than the surrounding matter. A temperature would finally be reached at which the still could no longer maintain its normal strength. The pressure inside the still would immediately cause a bulge at this point and the still would eventually blow up. The superheated liquid, petroleum, would flash in the fire box with a resulting ex-

plosion which would be disastrous. The industry attempted in a great many ways to overcome this difficulty, but without success. The life of a still runner was becoming shorter and shorter, and the terrain about the refinery was beginning to resemble no man's land in a movie set-up.

Dr. Burton of the Standard Oil Company of Indiana realized the importance of this situation and attempted to remedy it. He probably reasoned in somewhat this manner, that the factors involved in the reaction were not only pressure, temperature, and concentration, but also the raw material, a particular petroleum fraction, and the final products, gasoline, tar, coke, etc. Most of the other inventors had concentrated their attention on the type of apparatus in which the reaction was to be run and also upon a means of removing the coke as fast as it was formed. Dr. Burton, however, chose for his factor the raw material. He realized that there was a great difference in raw materials. He investigated this situation very thoroughly and finally found a type of oil which could be obtained in large quantities (evidently a paraffin base oil containing no aromatics) which on treatment in the regular manner produced very little or no carbon. This is not all that Dr. Burton did in this particular development, but it serves as an excellent example of the common method of attack used in the majority of our great industrial chemical developments. It might be said that Dr. Burton's cracking process was the first satisfactory commercial cracking process and maintained its paramount position for approximately seven years.

Another interesting example of research in an entirely different line follows this same method of attack. It has been known for some time that pure aluminum would withstand hot gases containing sulfur compounds. On the other hand, pure aluminum has very low tensile strength but would be an excellent thing if we were able to deposit a thin but continuous layer of pure aluminum on steel to be used for industrial purposes where strength and ability to withstand corrosion of certain types are necessary. Probably the most continuous metallic coatings we have today are those produced by the electrodeposition of metals. Unfortunately, aluminum cannot be deposited from the ordinary water solution due to the extreme activity between the aluminum metal and the water. Hydrogen is produced at the cathode. A literature search revealed the fact that a great many different combinations of salts in water solution had been tried in order to deposit aluminum

electrolytically but with no success. Some attempt was made to deposit aluminum from organic solutions with apparently no success.

The subject, however, interested some investigators at the University of Illinois, and it was their belief that the important factor in the reaction was the solvent, or electrolyte. The outstanding characteristic of an electrolyte for successful operation was that it must be free from replacable hydrogen and also have high electrical conductivity. Reasoning from atomic structure, it was thought possible to make a satisfactory electrolyte by using a tetra-alkyl-ammonium-halide in combination with an aluminum-halide. It was also suspected that a compound similar to the Grignard reagent in which the magnesium had been replaced by aluminum might prove satisfactory. These two types of compounds were tried out, and still hydrogen was given off. It was quite evident that the hydrogen did not come from the compounds but from some water present. As the compounds themselves were quite dry, the water must have come from the air above the electrolyte. This air was dried and the reaction, the electrodeposition of aluminum, took place on a copper cathode. The deposit seemed to be a fairly pure aluminum of very fine structure, and adhered fairly well to the copper below.

Another interesting type of industrial chemical research is the development of a new and more satisfactory chemical product to take the place of an old product. An example of this type of development is the well-known rayon, the comparatively new synthetic textile resembling natural silk. Again it is quite possible to outline the common method of attack somewhat as follows:

1. A list of the faults of the old products.
2. A list of similar products not now used for this particular purpose.
3. A selection from list No. 2 of the most suitable product from an economic and common sense standpoint.
4. Modification of this product to eliminate as far as possible what faults it has.

Tincture of iodine is a highly unsatisfactory household anti-septic when applied to open wounds. The content of iodine (seven per cent) is far too great and in many cases it probably does more harm than good because of its tendency to attack living tissue. The solvent alcohol (concentrated) is about as satisfactory a substance to put on a cut as a red hot iron would be. Lastly, tincture

of iodine is usually sold in a bottle with a cork or rubber stopper. Such a package is not exactly a satisfactory thing to carry in one's traveling bag or vest pocket. In brief, this antiseptic popularized by the World War is far from satisfactory for household purposes.

It is well known that both iodine and chlorine have powerful bactericidal action when in the free state. Weight for weight, these halogens are at least seven hundred times as powerful as the corresponding weight of phenol or carbolic acid. It would seem, therefore, that all that is necessary to make a satisfactory household antiseptic is to have a solid compound which when thrown into water would produce dilute solutions of iodine or chlorine (without injurious by-products).

This subject interested some investigators at the University of Illinois, and they decided that the type of solid compound which would prove most satisfactory would be a hypochlorite. After a rather extensive investigation, it was found that a fairly pure calcium hypochlorite containing some excess calcium hydroxide was fairly satisfactory. The liberation of chlorine when this powder was placed in warm water was not rapid enough to satisfy the investigators, so powdered dry aluminum sulphate was added with the calcium hypochlorite. This salt of a weak base produced, in the water solution, free hypochlorous acid which decomposed into chlorine more rapidly and gave quite satisfactory results. Incidentally, an antiseptic of this type can be taken in the form of a dilute solution as a gargle and will have genuine antiseptic properties, something that cannot be said of the majority of mouth washes.

Perhaps the most interesting type of research in applied chemistry or chemical engineering is the development of a new type of machine to do a particular unit operation more efficiently than any standard machine has done it previously. This type of research also can be outlined in a general sort of way as follows:

1. A list of all the basic requirements of the particular unit process.
2. A list of all apparatus used in other industries for a somewhat similar purpose.
3. A selection from list No. 2 of the apparatus which most nearly meets the basic requirements of the process.
4. Modification of this apparatus to meet the special conditions.

Examples of this type of research are very numerous. A few of the outstanding ones might be mentioned: the use of the Haber fixation of nitrogen machine for the manufacture of methanol; the use of mining and metallurgical equipment in the sugar industry; the use of the alcohol fractionating columns in petroleum refining; and the use of the X-ray machine, which formerly has only been used in surgical work, in a thousand different industries.

An interesting recent development at the University of Illinois is the utilization of the homogenizer and colloid mill in the manufacture of acetic acid by the catalytic partial oxidation of ethyl alcohol. It can be predicted, without fear of contradiction, that these two types of equipment will eventually be used in a great many different liquid-gas reactions.

In conclusion, the author wishes to reiterate that modern industrial chemical research is not a mysterious art, but is a highly developed scientific subject. The methods used are almost the same the world over, and the element of luck, though still of great importance, is becoming less and less significant with the passing of time. It might be said that any result desired in industrial chemical research can be had. It is only a question of money, men, and time.