INSTALLATION FOR AUTOMATIC ATTACK OF MATERIALS WITH A VIEW TO THEIR CHEMICAL ANALYSIS

FIG. 1

FIG. 2
FIG. 3
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ABSTRACT OF THE DISCLOSURE

An automatic chemical analysis apparatus is provided with a container for holding the materials to be tested, automatic filling, heating and mixing means all located externally of the container and a container cleaning arrangement which is provided with a device for completely inverting the container to discharge the material therein and to permit washing and drying of the container. The container may be located at a fixed station or indexed from one station to another for the performance of the various operations.

The present invention relates to a novel installation for the automatic attack of materials by suitable reagents to put them in solution or condition them with a view to further operations and, particularly, their chemical analysis.

The present trend toward automation of industrial processes is a well known phenomenon. In all cases, the automatic mechanisms must receive data on the quantity of the materials undergoing change. Very often this data is made up of the amount or amounts of materials in one or more of given constituents. The basic conditions for embodying automatic mechanisms lies then in the possibility of making the very process of chemical analysis automatic.

Processes of chemical analysis are numerous and varied. They include colorimetric, polarimetric, photometric, chromatographic, etc. and the means of embodying them to make them automatic differ.

However, in a great number of cases, analysis is carried out on a solution of the material to be analyzed in a liquid. Putting of the material to be analyzed in solution is carried out by an attack on said material by one or more acids or by any other chemical compounds that can assure that the constituent or constituents of the material to be analyzed are put into solution.

The material to be analyzed can be in two distinct physical states:

(a) In solid state—the material being, if need be, previously reduced by grinding to a powder state of the fineness necessary for a good homogeneity of the powder and for the possibilities of satisfactory and rapid attack, these conditions of fineness being able to vary from one material to another; or

(b) In a slurry or in suspension in a liquid—the solid material, already in the powder state, can be in the form of a suspension in water or any other liquid. This presupposes that the conditions of necessary fineness preexist or, if necessary, have been previously obtained by any suitable means.

If it is desired to make the analysis operations automatic, the first operation to be performed is to take a test portion from the sample of the material to be analyzed. This portion can vary from one material to another, of the order of magnitude of 0.5 to 5 gr., for example.

If the sample is in the form of a powdered solid, the test portion will be portioned out by a preset automatic balance to take the test portion from a given mass with a given relative precision.

If the sample is in a liquid suspension of slurry, the test portion will be taken as a given volume with a precision already predetermined. Knowledge of the solid mass contained in the volume of the test portion is necessary for making the chemical analysis.

It is then possible to regulate automatically the concentration of the sample so that a known solid mass corresponds to a known volume of the liquid suspension, to proceed to an automatic measuring of this concentration by automatic weighing of a known volume of the liquid suspension or to proceed to measuring this concentration by any other suitable means such as, for example and in a non-limiting way, a colorimetric measurement, an absorption measurement, etc.

In both cases mentioned above, that is, dry powder and powder in suspension in a liquid, present processes permit taking an automatic attack on the constituents by suitable reagents only when the solid phase is of slight hardness and slightly abrasive and when it is easily soluble in a rather slightly corrosive liquid, leaving only very little or no insoluble residue.

In all other cases, present processes are inadequate, since the results obtained are falsified by the appearance of residues that clog the apparatus and thus prevent the circulation of the products to be analyzed. On the other hand, the products treated cannot contain abrasive materials that damage the apparatus during their movement.

The present invention therefore has for its object a novel apparatus for automatically putting into solution or providing the desired conditioning of products to be treated or analyzed with a view to measuring, in a moist manner, the concentration of one or more constituents in the powder or the liquid suspension form. These constituents can be present in a solid phase, can be abrasive or not, can be of any hardness, and may be attacked by a very corrosive liquid that may or may not leave an insoluble residue that may or may not be abundant.

The invention therefore relates to the treatment of any product and particularly of ores in powder or in a liquid suspension wherein it is proposed to portion out one or more constituents. The reagents necessary for the attack may be concentrated inorganic acids or any other slightly or very corrosive product.

The product to be attacked can, therefore, contain grains of very different natures, even very hard and abrasive ones, such as quartz, and putting the constituents, which is to be portioned out, into solution can, if desired, be compatible with the existence of an insoluble attack residue that is even abundant and abrasive.

According to the invention, the apparatus for putting various materials, even very abrasive ones, into solution and conditioning them, with a view to further operations, and particularly their chemical analysis, is characterized by the fact that it comprises in combination: a control unit; one or more means operated by said unit to pour in one or more suitable containers given amounts of the product or products to be treated, one or more means controlled by said unit to introduce into said container or containers one or more reagents, one or more means operated by said unit to heat said container or containers for certain periods, one or more means operated by said unit to homogenize the solutions obtained, one or more means operated by said unit to send the solutions obtained to the analysis or evacuation means and one or more means operated by said unit to rinse said container or containers.

The characteristics and advantages of the present invention will be brought out in the following description.
given with reference to the drawing attached to this text which shows, by way of example, an embodiment and one of its variants.

In the drawings:

FIGURE 1 shows a diagrammatic view in elevation of the novel apparatus;

FIGURE 2 shows the diagrammatic view in plan of the apparatus;

FIGURE 3 shows the electrical control diagram;

FIGURE 4 shows a diagrammatic view of a variant of the apparatus of FIGURE 1, and

FIGURE 5 is a diagrammatic representation of the control circuits in the form of units.

For greater clarity, each of the elements making up the novel installation for automatic attack of the products to be treated has been represented by diagrammatic units. It is obvious that each of them may be in various forms and present any characteristics of capacity, power or sensitivity.

To facilitate understanding the invention, the apparatus will first be described with reference to FIGURES 1 and 2, then its operation will be explained with reference to FIGURE 3 and finally, one of numerous variants will be explained with reference to FIGURES 4 and 5.

FIGURE 1 supports various members 3 to 8 of the apparatus. It also contains the control mechanism of central member 11 which assures the successive movements of a container. Test tube 9, for example, is carried by arm 11a which moves it into cooperation with the various means of the apparatus to achieve automatically each of the operational phases necessary for the treatment from the receipt of the product to be treated to its removal after treatment.

Driving of member 11 can be achieved by any suitable well known means. A motor 4, controlled by control unit 12, drives member 11 by various mechanisms placed under the control of clutches.

According to the embodiment chosen by way of example, driving of member 11 is performed by means of motor 4 whose feed circuit is controlled both by a series of contacts operated by cam 14 and 15 mounted on the motor shaft (only contact C4 has been shown, for greater clarity) and by the circuits of a unit E4 (FIGURE 2) which is itself controlled by the circuits of units E2, E3 and E5 to E8, respectively, connected to units 2, 3 and 5 to 8. These circuits shown in the main diagram, FIGURE 3, will be described when the operation of the apparatus is explained.

Test tube 9, carried by arm 11a, is made of a material that can resist the conditions of use, for example, fused silica. Test tube 9 can have a different shape and contain, for example, from several cc. to several hundred cc. At the beginning of a cycle, test tube 9, already emptied and cleaned, is brought below distributor tube 2. It then receives the portion of the sample to be treated.

It is obvious that this distributor, which has not been shown, can be of any type. It can be used to proportion materials in powder form whose constituents are to be analyzed. It can also distribute these materials in the form of a liquid suspension or slurry. In the first case, the sample in the form of a powdered solid is distributed by a preset automatic balance to take out a certain mass with a certain relative precision.

If the sample is in slurry form, the distributor makes it possible to take out a predetermined volume with a certain precision. Thus, it is possible to collect a sample of known weight or volume in test tube 9.

The distributor apparatus is connected by wires 2 (FIGURE 2) to unit E2 which makes it possible to start the cycle of the auto-attacker by manual starting contact 16. E2 is connected to either the dry product distributing balance or slurry distributor and for this purpose has a switch 18 placed either at B or P, depending on whether the distributor used is the balance or slurry distributor. This connection makes possible automatic start-

of the cycle when the distributor selected has completed its operation.

Means 3 makes it possible to deliver predetermined proportions of reagents for chemical attack, dissolving or diluting.

This means can be made up of a burette with electrovalves made of materials that resist the conditions of use, such as glass, stainless steel, polytetrafluoroethylene, etc., these materials depending on the requirements of the distributor used.

A tank, not shown, feeds distribution pipette 3 by tube 10 and electrovalve EV1. When the pipette is filled to the desired level, this level being detected by platinum electrodes 25, a signal makes it possible to control closing of EV1. When the circuit of unit E3 goes into operation, electrovalve EV2 releases the reagent contained in pipette 3 and the reagent flows into test tube 9.

Means 5 is intended to permit either heating or cooling, as desired, of the attack container or containers to bring the products to a certain temperature for a determined time.

Heating can be obtained by any procedure, for example, by electric resistances. Cooling can also be obtained by using jets of cold air or cold water or any cooling fluids.

Means 6 is an agitation and homogenization apparatus intended to facilitate chemical attack and make the suspensions of solids uniform in the liquids contained in the attack test tube. This apparatus can be of any type, the agitation or homogenization being able to be obtained by vibration of the attack tube or by bubbling of a gas such as air, for example, in the liquid or in the suspension of the solid in the liquid or gain by using a magnetic agitator.

First of all, test tube 9 can receive a certain amount of water, for example, which is distributed under conditions identical with the distribution of the reagents by electrovalves EV1 and EV2 through electrovalve E6 (FIGURE 2).

A hood, not shown, and a ventilation circuit working from the time the heating starts until planned removal at unit 7 make it possible to eliminate harmful or corrosive vapors.

Means 7 which is used to remove products thus treated has a tube 22 made of a material that cannot be attacked under conditions of use. It can be attached to the attack container, or be stuck into it at the right times on a suitable mechanism such as a rack in the embodiment chosen by way of example.

Means 7 has a motor operated by the circuits of unit E7. It drives, by a pinion, a rack to which is attached removal tube 22 which can thus go down into test tube 9 to make the desired removal.

A control contact 7a, operated by the rack at the end of its path, makes it possible to stop the motor. Reversal of the direction the motor runs, makes possible the raising of tube 22. A second control contact 7b makes it possible to stop the motor when the rack reaches its upper limit position.

Means 8 makes possible the cleaning of the attack container. It can involve a simple emptying container, test tube 9 being made to swing to pour its contents into funnel 8. Emptying can also be carried out by suction or any other means. A device for automatic rinsing by a jet of liquid, hot or cold water, can be added to it, this rinsing being, if desired, followed, by a drying by any process, for example, blowing of hot or cold air.

The test tube can be turned upside down during the movement of arm 11a from unit 7 or unit 8 by arranging on arm 11a, which can turn around its horizontal axis, a pinion which engages in toothed track 11b arranged between unit 7 and unit 2. The test tube makes a half-turn by passing from 7 to 8 and goes back to its normal position by traveling the path from unit 8 to unit 7.

Performance of the various operations carried out with the apparatus that have just been described is controlled by programmer 12 made up, as shown in FIGURE 2, of a certain number of units, some of which are identical.
Each of units E3, E5, E6, E7 and E8, attached to units 3, 5, 6, 7 and 8, have a timing device T making it possible to regulate the length of each operational phase at will. Each feeding is provided by unit E1 when its switch 13a closes input circuit 13. Since transformation to direct current can be realized by any type apparatus, the diagram of unit E1 has not been shown. The main circuit of FIGURE 3 carries only references B1, B2, designating input terminals of the direct current provided by E1 and lines A1, A2 distributing the alternating current.

Unit E0 relates to the device for detecting the levels of the liquids in the distribution pipettes and for controlling the distribution electrovalues. It works with unit E3.

The particular role of the circuits of unit E4, controlling the passage from one operational phase to the following, has been indicated on the drawing by the presence of signal light V4 which lights up, as will be seen during the description of the electric diagram either in case a timing device T does not function thus running the risk of interrupting the cycle between two operating phases or when the apparatus is faulted.

Operation of the apparatus will be explained with reference to the electric diagram shown in FIGURE 3.

At the start, arm 11a, carrying test tube 9, is in the direction of loading unit 2.

Contact C2, operated by cam 14, is therefore closed and contacts R3A, R3B, operated by cam 15 (FIGURE 1) are pivoted, i.e., occupying position ac.

15A therefore cuts off feed of the motor which was made by the following circuit: line A1—motor 4—contact 15A in position ab—contacts R3A and R5A in position end 15A, A2.

15B connects line A1 to signal light V4 which, outside of the particular starting position, means that an operation timing circuit is out of order. The feed circuit of V4 is the following: line A1 contact 15B at ac—light V4—contact R4A at ac, wire j9, contact R3A, R5A, line 11a.

As was seen above, it is possible to make an analysis either from dry products or from products in slurry form. Depending on the case, switch 18, shown in unit E2, FIGURE 2 and in electric diagram FIGURE 3, should be either in position B to route the control signals, produced by generator 20, to a distributing balance 21 with a determined weight of dry product or in position P to control the slurry distributor 19.

In this latter case, for example, the loading of test tube 9 can be made upon closing of contact C2 by cam 14, i.e., when test tube 9 is facing loading unit 2.

In case the dry products are to be removed, switch 18 facing stud B, it can be seen that contact C2 this time controls starting of unit 20 intended to provide a series of pulses necessary for operating distributing balance 21.

At the end of loading, distributor 19 or 21, whichever is used, closes contact 17 or 23 intended to operate relay R2 by starting the following operation by applying voltage to motor 4.

Actually, in both cases, relay 2 is energized by the following circuit: terminal B1, contact 17 (or 23) in position ac, relay R2, terminal B2. Light V2 goes on and contacts R2A and R2B close, making possible:

1) Holding of relay R2 by circuit B1, contact R3B in position AB, contact R2B keeping relay R2 and B2 closed and
2) Operation of driving motor 4 by the circuit line A1, motor 4, contact R2A in position AC, line A2.

A contact 16, placed parallel with contacts 17 and 23 of apparatus for distributing the products to be treated, and mounted on unit E2, makes possible manual control of the starting cycle.

The rotation of motor 4 causes the rotation of shaft 11 and cam 14 which by lifting the control strip of contact C2 breaks the control circuits of the apparatus for loading the product to be treated.

Also upon rotation of cam 15, contacts 15A and 15B cause, on the one hand, normal reestablishing of the feed circuit of motor 4 by the circuit: terminal A1 motor 4 terminals a and b of contacts 15A, R3A and R5A, terminal A2; on the other hand, turning off of light V4 by opening contact 15B.

In its rotation, arm 11a brings test tube 9 to acid distribution unit 3. Cam 15 again causes pivoting of contact 15A which cuts off the energizing circuit of the motor by taking position AC.

However, although contact 15B occupies position AC, light V4 does not go on because relay R4 is energized under the control of contact R3C of relay R3 and transfers its contact R4A into position AC, thereby opening the circuit of light V4. Relay R3 is then actually fed by closing of contact C3 under the action of cam 14 and continues to be fed under the action of a timing circuit. Since this circuit can be of any type, it has been represented by unit T3 and for ease of explanation, the excitation coil of relay R3 is designated by r3 and its holding coil by R3. It is obvious that two separate relays could also have been chosen.

The feed circuit of r3 is the following: terminal B1, closed contact C3, coil r3, terminal B2. Relay R3 continues to be fed by the following circuit: B1, contact r3A in closed position, the circuit of timer T3 maintaining the feed of relay R3, R3 and terminal B2. It is clear that r3 can also be a relay independent of R3, and that the timer can be any type, since connection 1 does not depend on the type selected.

When relay R3 is energized, contact R3A passes to position AC, opening the motor feed circuit. Contact R3C causes feeding of relay R4 by the circuit: terminal B1 contact R3C in position AC, relay R4, B2. Shifting of the contact R3B to position AC cuts off the holding of starting relay R2, energized at the end of the loading operation.

Finally, contact R3D passing from position AB to AC causes operation of electrovalueOf EV2 by circuit line A1, coil of EV2, R3D, line A2.

Distribution pipette 3, containing a predetermined volume of acid, empties into test tube 9.

When the period set on timer T3 (FIGURE 2 and FIGURE 3) has been completed, relay R3 is de-energized so that its contacts resume position AB. However, although contact R3C, by coming back to its rest position, cuts off the feed circuit of relay R4, this latter is kept energized by the discharge of capacitor 24 which was charged during feeding of R4.

Consequently, the following circuit is established and permits starting of motor 4 to carry the test tube to loading unit 3: line A1 motor 4, contact 15A always in controlled position ac, wire j1, contact R4A always in controlled position, wire j9, contacts R3A in rest position ab, line A2.

The circuit between line A2 and contact R5A has been shown as cut by wavy lines to indicate that there are other control contacts playing identical roles but which have not been shown to make the drawing less crowded.

Also each timer T is completed by a signal light V placed parallel with relay R which it holds. A light placed on corresponding unit E permits signalling of its functioning.

When motor 4 again starts under the control of contacts R3A, contacts 15A and 15B return to the position shown in the drawing until arm 11a brings test tube 9 to heating unit 5.

After the discharge of capacitor 24, since relay R4 is no longer fed, it releases its contact R4A which comes back to the rest position.

It should be noted that if, as a result of the failure of timer T3 to function, relay R3 is not energized, relay R4 cannot be fed so that light V4 goes on by the following circuit: line A1 contact 15B in controlled position ac, light V4, contact R4A in rest position ab, wire j9, contacts R3A and R5A in rest position ab, line A2.
It is obvious that light V4 can be replaced by any other alarm device, acoustic, for example, and cause any desired operation depending on the operation in progress.

The role played by contact R3A in coming back to its rest position has been seen. During starting of the motor, to be held in contact to R3D to its other position, ab opens the coil of electrovalve EV1 intended to fill pipette 3 to a predetermined adjustable level. The following circuit is established: line A1, coil of electrovalve EV1, contact R3D at ab, line A2. This control permits filling of pipette 3 which contains two platinum electrodes 25, one going to the holder of the pipette and the other coming to an adjustable and predetermined level. When electrovalve EV1 is operated, the acid goes by a filling orifice to the lower part of the pipette. When its level reaches the upper adjustable electrode, an electric signal is sent by wire 110 to break the circuit of the coil of electrovalve EV1 so that pipette 3 is ready to deliver the same amount in the next cycle.

A light V6 permits signalling of a failure in filling the distribution pipette (lack of reagent in the tank, flow hole stopped up) or again the failure of the liquid detection relay when the liquid level drops. For this purpose, the circuit of light V6 is broken only by deenergizing of a relay which occurs only after detection of the level by electrodes 25.

When arm 11a brings test tube 9 into oven 5, contact C5, operated by cam 14, closes the feed circuit of relay R5 whose holding is assured by an adjustable timer T5. When relay R5 is fed, it causes the shifting of its various contacts which in turn control the starting of heating resistances and of ventilation. Heating is cut off at the timing when R5 is no longer fed, but, on the other hand, ventilation continues until the removal operation, due to a self-feeding circuit which is maintained until the opening of a contact of relay R7 of the circuits attached to the removal device.

So as not to overload the drawing needlessly, relay R5 has not been shown, since its feed circuit is the same as that of relay R3. However, contact R5A in the lighting circuit of light V4 in case R5 does not function has been represented, as a reminder. In case of a normal functioning, a contact R5C, placed parallel with contact R3C, causes energizing of relay R4 whose role has already been explained in connection with the starting of motor 4.

After the heating operation, the test tube is brought to the unit for filling of water and agitation. Filling is performed with two electrovalves in a way identical with that already described in connection with acid unit 3. The agitation device, one of the contacts of relays R6, controlled by contact C6, is used. Operation of relay R6 and its timer are identical with that of relay R3, the control circuits of R6 have not been shown so as not to overload the drawing.

At the end of the operation, motor 4 moves test tube 9 to removal unit 7.

This unit has a metal box 7 inside of which is placed a reversible motor 7m whose shaft is provided with a pinion. Motor 7m drives a rack supporting a tube 2d connected to the colorimetric proportioning apparatus, not shown. This tube is intended to remove the treated product from inside test tube 2.

Operation of this motor is performed by means of two contacts. One is operated when the rack is at its low position, the other when it is at its high position. These contacts 7A and 7B (FIGURE 1) are normally closed and are connected in series in the feed circuit of this motor. They make it possible to break this circuit to limit the range of movement of the rack.

A third contact 7C, opening at the time of the descent of the removal tube and closing at the high position, permits starting of motor 4 only when the removal tube has completely come out of the tube.

Thus, when contact C7 is operated by cam 14, the feed circuit of relay R7, not shown, is closed. This relay, kept fed by its timer T7, operates its contacts, one of which closes the feed circuit of motor 7m. This motor then causes the descent of the bar which, when it reaches the low position, opens contact 7a and cuts off feeding of 7m.

When the period of time set on T7 has been completed, relay R7 ceases to be fed, causing an order to be given for the bar to contact 7b and move to the opposite position. The bar goes up and reaches its high position, opening contact 7b and closing contact 7c. Contact 7b breaks the feed circuit of motor 7m as explained above.

To obtain perfect reliability in functioning, the circuit for restarting motor 4 is no longer operated, as in the preceding case, but only de-energized of the corresponding relay 7R at the end of the timing and by the contact of relay 4 kept fed by the discharge of capacitor 24, but by closing of contact 7C.

Motor 4 then causes test tube 9 to pass from unit 7 to rinsing unit 8. During this movement, arm 11, which can turn around its horizontal axis and which has a toothed part that can mesh with toothed track 11b, arranged between units 7 and 8, comes in contact with track 11b. Arm 11a therefore turns both around the vertical axis of rotation and its horizontal axis, thus progressively bringing test tube 9 by pushing it under tube 2.

Contact C8, closed by cam 14, closes the feed circuit of relay R8, held by its timing device T8. This circuit obviously has not been represented because of its identity with those of unit E3 already described. One of the contacts of relay 8 operates the rinsing electrovalve so that, at the end of the timing, test tube 9 is ready to receive again the product to be treated at unit 2.

For this purpose, the return of contacts R8 to rest position causes restarting of motor 4 by contact R4A. Arms 11a, meshing with track 11b, completes the return of test tube 9 by placing it under tube 2.

Thus, it is easy to see the importance of this novel apparatus since it is possible to treat products, it being possible during the treatment to bring about precipitates which, due to the devices used, no longer impede the circulation of materials as in previous apparatus.

Another advantage of the apparatus is that it makes possible the independence of the length of each operating phase and the checking of the good functioning of the apparatus used in each of them.

Finally, the apparatus makes it possible to work on products of a very diverse nature without requiring any transformation at all, the periods of each phase merely having to be regulated, depending on the reactions to be obtained.

It is obvious that the present invention is not limited to the simple embodiment just described and that it would not be going outside the scope of the present invention by providing, for example, other reagent distribution units either before or after thermal treatment, the rotating member being able to have as many containers as there are operating units.

Also the timing circuits, which can be made up of transistor circuits controlling the discharge of a capacitor through variable resistors, could be replaced by a single pulse distributor delivering for each operating phase a number of pulses previously recorded in a memory of any type and particularly in those of an electronic computer of any type.

In other words, it would not be going outside of the present invention to control the apparatus with an electronic computer where output pulses from the computer would control either directly or by a relay the various circuits relating to starting the motor or starting the operations of distributing reagents, heating, removal or rinsing, since the pulses thus available play roles equivalent to the various commands resulting from each of units E. Moreover, to avoid having to start motor 4 several times during the same cycle, it would be possible to provide one or more one-turn clutching devices, the simple command of one or more clutch magnets making it possible to cause arm 11a to pass from one unit to the other.
Finally, detection of anomalies can be assured by various devices: thermal switches for heating anomalies, float switches for detecting the level of the liquid, devices for checking the time period in the case of an anomaly in the performance of certain phases, etc. These various devices can trigger the stopping of the cycle with the automatic return to the rinsing unit and elimination of the intermediate phases.

To illustrate the possibilities of applying the invention to various embodiments, reference is now made to FIGURE 4 which represents a preferred variant of the invention.

According to this variant, the apparatus operates according to the same principles as those that have just been described and with similar means. However, the present construction utilizes features that give the apparatus qualities of increased reliability, rapidity, output and flexibility while reducing its size and cost.

On the other hand, elimination of mobile elements makes it possible to reduce the time of maintenance of the apparatus, while the control circuits are considerably simplified.

Moreover, the lower part of the attack container is placed in a heating apparatus so that heat losses and the length of operations are reduced.

Looking at FIGURE 4, container 31 can receive a tube 33 containing the liquid the apparatus 33 of any type and able to deliver, for example, a determined volume of the product to be treated in slurry form or else to deliver a predetermined weight of the product in powder form. This distributor is electrically connected to control unit 34 by means of electric cable 35. It is clear that, depending on the type of distributor 33 used, conduct 32 can be eliminated.

Container 31 receives reagents suitable for attacking the product to be treated by means of tube 36 which goes into the neck of the container. The other end of the tube is divided into two branches 37 and 38, one leading to electrovalve 39, the other to electrovalve 40. These electrovalves are, respectively, connected to control unit 34 by electric cables 41 and 42. Tanks 43 and 44 contain reagents serving to fill flasks 45 and 46.

Finally, the upper part of container 31 is covered by bell 47 collecting the gases and vapors produced during the operation. These vapors, channeled by conduit 48, are evacuated by fan 49.

The lower part of the container is surrounded by an electric oven 50 controlled by unit 34 to which it is connected by electric cable 51.

A magnetic agitator, intended to homogenize the solutions, has an apparatus 52 creating a variable field to make plate 53, resting on the bottom of the container, vibrate. Apparatus 52 is connected by electric cable 54 to control unit 34.

A suction apparatus 55, controlled by unit 34 to which it is connected by electric cable 56, makes the necessary removals from the base of container 31 which, for this purpose, has a tube 57. The samples, thus removed, are directed toward the analysis apparatus by tube 58 under the action of the command coming from unit 34. If the container can be tilted, 57 can be eliminated.

At the end of the operation, the apparatus is rinsed with water, or possibly a suitable mixture, brought by tube 60, whose flow is controlled by electrovalve 61, which is operated from unit 34 by cable 62. Emptying, therefore, is made under the control of control apparatus 34 and suction organ 55, by tube 59.

Three lights V1, V2, V3, which light up, respectively, when the apparatus 33 is being treated and the reagents are lacking in distributor apparatus 33 or in flask 45 or 46 have been represented, as a reminder, at unit 34. Other lights and a suitable complete warning system can be provided, particularly in the case of power failure, for example.

Beneath these lights, are present by way of example, five regulating buttons t1 to t5 making it possible to set the time period of the various operating phases and to start one or more phases.

The electric circuits of control unit 34 have been shown, FIGURE 5, in a diagrammatic form, for the sole purpose of facilitating understanding of the operation of the apparatus. To keep the diagram in all its generality, each circuit unit has been drawn as a rectangle, these circuits actually being able to be any type and adapted to the techniques used.

Lines t1 and t2 are connected to a suitable current source. Unit C1 represents a circuit unit serving to control relay R1, starting the operation of product distributor 33, FIGURE 4. In its simplest form, C1 would contain only a simple manual control button which by its closing would cause energizing of relay R1. This relay obviously operates a certain number of contacts, one of which, placed in the circuit unit shown by T1, causes its holding during the period set by corresponding regulating button t1 shown in FIGURE 4.

In fact, unit T1 need not have timing circuits, since a contact at the end of the operation of the product distributor can break the holding circuit of relay R1, if this relay is used, and establish, for an instant, circuit t1-T1-C1-R1-C1-R1.

Relay R2, on being energized, controls the opening or closing of its contacts. One of them, placed in unit T2, permits starting of the other which will keep relay R2 energized during the entire period set by button t2. Another contact of R2 opens electrovalve 39 whose opening causes the flow of a certain proportion of reagents into container 31 through tubes 37 and 36.

When the timing circuits of T2 come to rest, one of them starts unit C2 which in turn causes energizing of relay R3, one of whose contacts operates electrovalve 40.

Since circuits C2, R3, R1 are similar to the preceding circuits, they and intermediate rank circuits 4, 5 and 6, have not been shown on the drawing.

After the introduction of the appropriate proportion of reagent contained in 46, relay R4 is energized.

Since this relay pertains to the heating control, control circuits C4 of relay R4 can be made up of contacts of one or more thermostats regulated so that the oven keeps a fixed temperature. Timing circuit T4, whose holding period is set by t4, makes it possible to start energizing of relay R4 operating magnetic agitator 52.

It should be noted that it would have been possible to arrange the control of energizing of relay R4 corresponding to reagent distribution by the circuits of timer T5, to energize relay R5 simultaneously. In this case, the removal operation, controlled by relay R6, can be started by timer T6, i.e., after a period of time fixed by regulating button t6 and not by means of regulating button t5.

Relay R6 controls suction apparatus 55 and routing of what has been removed which is brought by tube 57 to tube 58 which leads to the analysis apparatus. After this operation, timer T7 starts energizing of relay R7 by control circuits C7.

Electrovalve 51 is then operated by closing of a contact of relay R7. The washing water flows by tube 50 into container 31 while the residues carried by the flow of water are sucked by organ 55 and evacuated by tube 59.

Stopping occurs after a period set by button t5 which has not been shown, to facilitate reading of the drawing. The circuits of timer T7 are such that the suction is prolonged for a short while after closing of electrovalve 51 so that container 31, completely rinsed and dried, can receive a new sample of the product to be treated. For this purpose, the circuits of T7 cause the closing of a contact of C7, so that the operations are automatically repeated as just described.

These circuits are obviously given only by way of example. Control unit 34 could be replaced by a simple apparatus which transforms the pulses transmitted by a computer device having one or more preregistered programs, into pulses capable of controlling the functioning
or stopping of the various organs enumerated, this program or these programs being able to be automatically modified depending on the results of the analysis or depending on chance conditions such as power failure, lack of supply of reagents, etc., which may occur. Thus, due to the novel structure of the apparatus, it is possible to get maximum output from the flexibility of the electric control circuits since, due to the program, or simply by inserting removable connections between organs $C_i$ and $T_i$, for example, it is possible to control the performance of the operations in any order such as introduction of the reagent contained in flask $46$ before introduction of the reagent contained in flask $45$, but also to control several operations simultaneously, which is a result that could not be obtained by the first embodiment. Therefore, it is possible to control, depending on the products to be treated, the simultaneous introduction of the products and reagents and to control the magnetic agitator, if necessary, at the same time as the removal operation.

Further, distributor $33$ can have, according to the invention, a densimeter, which makes it possible to know the weight of the products to be treated that are contained in constant volume by the preparation introduced by this distributor into the container. The results furnished by the densimeter are then used to modify the volume of the reagents introduced into the container so as to proportion the weight of the reagents to that of the constituents. These results are transmitted directly or by control unit $34$ to the various organs involved and in particular to the timers involved. In case the volume of the reagents is checked by a suction or forcing syringe, the results provided by the densimeter are then used to extend or shorten the length of the mobile part of the syringe. The regulating device itself, which can be of any type, has not been shown on the drawing. Flasks $45$ and $36$ stand for all liquid distributing devices.

Although several embodiments of the novel apparatus have been described, it is clear that numerous other substitutions, additions or omissions could be made to the elements of the apparatus or to their combination without going outside the scope of the present invention. Thus, certain elements could even be mobile and be made to work automatically with the container, for example, automatic introduction of the tube channeling the reagents, or descent and withdrawal of the removal pipettes, etc. Also the rinsing and emptying devices could be modified so that these operations could be performed by tilting container $31$ after complete removal of tubes $36$ and $60$ and hood $47$. Finally, other elements could be added such as cooling means, for example.

Finally, several containers could be served simultaneously or successively by the same distributing organs. In case the products to be treated differ, it is possible to double all the timers or a part of them, while leaving in common the general circuits of unit $34$ and by providing a control relay starting, depending on the container used, either a first or second set of timers.

Having thus described my invention I claim:

1. An automatic apparatus for preparing materials for chemical analysis comprising container means adapted to receive the material to be prepared, material supply means for automatically supplying material to said container means, reagent supply means for automatically supplying reagents to said container means, temperature control means external of said container means adapted to automatically control the temperature in said container means, automatic mixing means for homogenizing the solutions obtained within said container means, removal means for automatically removing said solutions from the container means for chemical analysis, automatic cleaning means to prepare said container means for receiving a new charge of material including means for inverting said container means during operation of said cleaning means and control means for automatically operating said material supply means, said reagent supply means, said temperature control means, said mixing means, said removal means and said cleaning means according to a predetermined program.

2. An automatic apparatus as set forth in claim $1$ wherein all of said means are stationary.

3. An automatic apparatus as set forth in claim $1$ further comprising a conduit connected to the bottom of said container means, pump means connected to said conduit and valve means connected to said pump means and controlled by said control means to sequentially divert the material removed from said container means to diverse receiving means.

4. An automatic apparatus according to claim $1$ wherein said reagent supply means is provided with regulating means adapted to control the amount of reagent which is supplied to said container means.

5. An automatic apparatus according to claim $1$ wherein said automatic mixing means is comprised of magnetic means located in said container means and magnetic field creating means external of said container means and adapted to vibrate the magnetic means within said container means.

6. An automatic apparatus according to claim $1$ wherein said container means is movably mounted and further comprises means for automatically indexing said container means to the other mentioned means according to a predetermined program.

7. An automatic apparatus according to claim $6$ wherein said removal means comprises a pipette adapted to be lowered and raised into and out of said container means.

8. An automatic apparatus according to claim $1$ wherein said control means comprises a plurality of electrically connected control units, one of said units being associated with each of said means to be controlled and having timing means to determine the period of operation of the means to be controlled thereby.

9. An automatic apparatus according to claim $8$ further comprising a plurality of rotatable cam means and a plurality of switches adapted to be operated by said cam means to initiate the operation of said timing means.

10. An automatic apparatus according to claim $8$ further comprising means responsive to the termination of operation of each of said timing means to initiate the operation of the next timing means in a predetermined sequence.

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JOSEPH SCOVONNEK, Primary Examiner

U.S. Cl. X.R.

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CERTIFICATE OF CORRECTION

Patent No. 3,466,146 Dated September 9, 1969
Inventor(s) Jean F. Tabourin

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In the heading the assignees should be as follows:

(SEAL)
Attest:
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