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ON APPARATUS FOR PROMOTING THE INTERACTION OF LIQUIDS AND GASES.[1]

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BOTH in theoretical and industrial chemistry one of the most frequent operations consists in the interaction between liquids and gases. On the laboratory scale this operation presents but few difficulties and is generally treated rather briefly, both in chemical literature and in laboratory teaching. Even here the matter is not quite so plain as is assumed by many chemists, and the success or failure of analytical operations is sometimes intimately connected with this question. Where the task is merely that of treating a liquid with a gas, irrespective of the quantity of the latter consumed, the question is certainly simple enough. But where it is required to completely absorb every trace of a special gas diluted with a large quantity of inert gases, the ordinary wash bottles and similar apparatus are mostly insufficient. I have made this matter a special study in connection with the estimation of nitric oxide in chamber-exit gases and similar difficult cases, and I have described the best shape of absorbing apparatus for such purposes in a communication to the *Zeitschrift für Angewandte Chemie*, *1891*, p. 567, and in the *Journal of the Society of Chemical Industry*, *1890*, p. 1015.

The task becomes far more serious when operations on the

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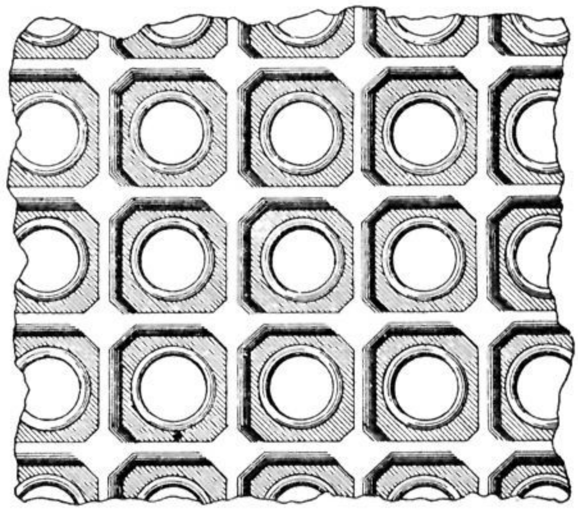
large scale are concerned. The principal difficulties encountered here are the following: The immense volumes of gases which generally have to be dealt with and which sometimes contain but a small fraction of the gas or vapor which it is essential to bring into reaction with the liquid, and the necessity generally existing of employing the least possible quantity of liquid and of obtaining a strong solution, at the same time depriving the gases as completely as possible of the active constituent in question. Special difficulties arise in many cases through the corrosive action of either the gases, or the absorbing liquid, or both; from the fact of tar, soot, flue dust, and other matters obstructing the channels through which the gas and the liquid have to pass; through the pasty condition of some absorbing media, such as Weldon mud, and in other ways. A whole book might be written on the construction of absorbing apparatus for all these cases, and if all the patents taken out in this line were to be noticed and illustrated therein, such a book would be of formidable dimensions.

Certain points have always been taken for granted in devising apparatus of the class we are now speaking of. Everybody has tacitly assumed that the efficiency of such apparatus will be in proportion to the surface of contact and to the time which is given for the action. Dr. Hurter has attempted to give a scientific theory of that subject in several communications published in the *Journal of the Society of Chemical Industry*, *1885*, 639; *1887*, 707; *1893*, 226. Unfortunately the various conditions to be taken into account are too complicated and partly too little known to be amenable to scientific treatment, and if such a treatment is attempted after all, it may sometimes end in a decided failure and lead to entirely erroneous conclusions, as I have shown in my criticism of Dr. Hurter's last paper (the same journal, *1893*, 417). I shall not detain you here with a repetition of that criticism, but I shall straightway come to that apparatus for effecting contact between gases and liquids which I have devised myself and which has been carried out by Mr. Rohrmann, my co-patentee, in a manner acknowledged on all sides (quite prominently so by Dr. Hurter himself) as being a hitherto unparalleled achievement in chemical pottery.

I believe I may assume that my apparatus, which I have styled "plate column" or "plate tower," is already known to many of my hearers both from my own publications and from the trouble my friend, Mr. H. J. Davis, has taken to bring it before the American public. It is also described in the second edition of my "Sulphuric Acid and Alkali," at least so far as it is used in the manufacture of sulphuric acid, and I must refer to this for a detailed treatment of the subject. I will, therefore, only say a few words about it and illustrate these by a diagram and by a small specimen of the plates.

In order to adapt my apparatus to every kind of chemicals, the apparatus is made of a description of stoneware, absolutely resisting any chemical action of the strongest acids or alkaline liquids at temperatures far above 100° C., being at the same time very little sensitive to sudden changes of temperature up to the same limit. From



Plan of Lunge-Rohrmann Plate.

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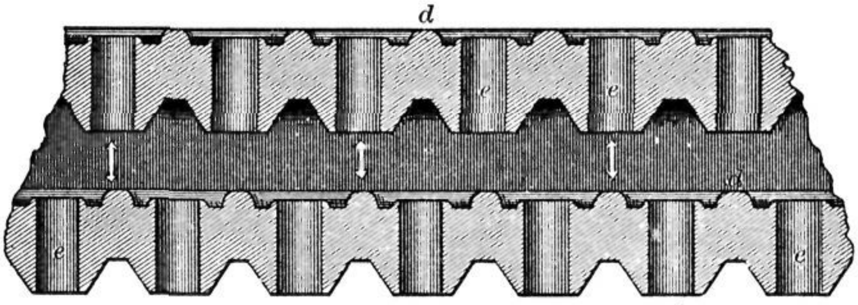

Section of Lunge-Rohrmann Plate.

this material my "plates" are manufactured on the following plan: Each plate is covered with a net work of ledges, about an eighth to a twelfth of an inch high, forming square basins of three-fourths of an inch square each, or less than that, according to the size of the holes, so that a plate of two by two feet contains from 800 to 1,200 of such tiny basins. Each of these is provided with a central hole differing in width from less than a quarter to half an inch, according to circumstances. The top of each hole is surrounded by a slightly raised margin about a sixteenth of an inch deep. The plates are made on two different patterns, in one of which the basins are distant from the edge by half the width of a basin, whilst in the other pattern they come right up to the edge. Consequently when these two patterns of plates are superposed over one another, the holes in each plate correspond to the center of the cross formed by the ledges of the four basins just above and below. Hence when a liquid is run upon the plates each of the tiny basins is filled independently up to the edge of the margins round the holes. It then overflows through the hole and the drop thus formed falls upon the cross of ledges underneath, and is scattered over the four corresponding basins. Furrows on the bottom sides of the plates prevent the drops from running along the bottom and falling down in other places than those where they are intended to get to. While in this manner the liquid is spread on each plate in a thin film, which is constantly renewed by drops from above in the most regular manner, the gases are just as regularly divided, and issue in about 1000 jets through each plate without the possibility of any false channels being formed which so frequently occur in coke towers. As the gas jets ascend through the holes in any one of the plates, they strike against the solid portions of the plate next above, and are thus deflected from their course and constantly mixed over again. All this will be more completely understood by looking at the diagram.

It is easy to see that ordinary pottery, even of the best known makes, will not stand in this case. The plates must be very thin and at the same time they must be absolutely level and true in all parts. They must also bear a certain mechanical strain and sudden changes of temperature together with complete resistance to the action of chemicals. I did not know of any chemical pottery in Europe which fulfilled these conditions as well as that of Mr. Rohrmann, and for that reason I associated myself with him in that matter. I am glad to say that the large specimens of his plates submitted to the London and

Liverpool meetings of the Society of Chemical Industry by buyers of the same have excited universal admiration there.

Where no metal, not even lead, can be tolerated, the plates are made of circular shape and are built up with stoneware rings to keep them in proper distance, in stoneware pipes, thus forming columns of circular section and containing up to forty plates or even more. The sectional area of these columns is limited by the practicable width of the stoneware pipes and does not generally exceed two feet six inches. Large streams of gases, for which such a section would not suffice, have to be sub-divided among two or more columns. Where, however, lead is not excluded, the plates are made square, and are combined by means of specially designed bearers in such manner that any sectional area can be produced, the whole being enclosed in a leaden shell. The bearers support each plate independently of the others, and cover the whole of the lead so as to protect it entirely against the action of the heat and even of the chemicals, so that this system might be employed even in such cases where unprotected lead could not be employed.

Plate towers of these two constructions have now been proved to be, size for size, probably the most efficient apparatus hitherto invented for bringing gases into intimate contact with liquids, except in such cases where there is some solid or tarry matter present which would obstruct the holes and fill up the small basins. In all other cases the plate towers are from ten to twenty times as efficient as an equal space of the best arranged coke towers; and when they are compared with empty spaces, such as vitriol chambers, their efficiency is found to be hundreds of times greater than that of those spaces. From the nature of the case, plate towers can not be made of cheap materials turned out at a low price, but they are after all far cheaper than other apparatus with which they enter into competition, and would be so, even if their initial cost were equal to that of the coke towers or vitriol chambers they replace, seeing that they require next to no space, foundations and buildings, and that there is no pumping of liquids to a great height.

I now beg permission to illustrate my assertions by a number of examples taken from practical cases. I shall not touch upon some applications which may ultimately become very important indeed, such as the manufacture of acetic acid, but I shall confine my remarks exclusively to the application of plate towers in the manufacture of the so-called heavy chemicals, that is, sulphuric, hydrochloric, nitric acid, and chlorine.

The application of the plate towers in the manufacture of sulphuric acid takes place in three different ways, as a partial substitute for the Glover towers, as a partial or entire equivalent of the Gay Lussac tower, and as a partial substitute for the vitriol chambers themselves, enabling a set of chambers to produce up to sixty or seventy per cent. more acid than without the plate towers. I shall quote practical instances for all three applications.

As far as the Glover tower is concerned, it stands to reason that it can not be entirely replaced by a plate column, and that for two reasons. In the first instance, the quantity of dust carried over from the pyrites, or even sulphur burners, is so great that the plates at the bottom of the tower would soon be choked up. Secondly, the temperature of the gases entering at the bottom and that of the acid descending in the tower is too different to expect any kind of pottery to stand the inevitable sudden changes and inequalities, at least in ordinary cases. Where, however, both these objections are done away with by a previous utilization of the burner gases for concentrating acid in lead or platinum dishes, as in Henry Glover's plan ("Sulphuric Acid and Alkali," second edition, **1**, p. 201), the whole Glover tower might be made of a plate column. In the ordinary towers, however, the bottom part will have to be packed in the usual way with a network of bricks, or flints, or cylinders, while the upper part is filled with my plates. Or else the same plan may be adopted which has been applied with full success by Dr. Petri at the Buchsweiler works in Alsace. There they burn spent gas oxide containing, besides free sulphur, some ammonia salts, part of which is not removed by washing and exerts a destructive influence in the niter in the Glover tower. This well-known reaction by which the nitrogen of both the ammonia and the nitrous vitriol is set free as such, takes place only at high temperatures. Reasoning from this, Dr. Petri put on the top of his ordinary Glover tower a small plate column, consisting of thirteen layers of two plates each. The gases issuing from the Glover tower enter this small column at a temperature of 90° and leave it at 60° C., while the fresh nitrous vitriol is run in at the top and runs away at the bottom in order to feed the Glover tower. In this small column, holding altogether forty cubic feet, eighty per cent. of the denitration takes place, for which an ordinary Glover tower of eight by eight by twenty-five feet, equal to 1,600 cubic feet, is generally provided, leaving only twenty per cent. of the denitration to the Glover tower and effecting the denitration at such a low temperature that the ammonia exercises no injurious action. The contrast between the efficiency of those forty cubic feet of plate tower and the 1,600 cubic feet of ordinary Glover tower space is certainly startling. If, by the way, some of the plates should crack, they would all the same remain in their places and perform their work nearly as well as before.

The action of the plate column as a Gay Lussac tower is illustrated by an American works where a set of chambers of 212,000 cubic feet is provided with a plate tower of twenty layers of ten plates each. This tower, possessing about 280 cubic feet of active space, now fulfills the function of a coke tower of about 4,000 cubic feet. But it is evidently too wide for its present work, and hence requires too much acid for feeding it equally all over. It is hence intended to pass the gases from another set of 123,000 cubic feet through it, and it is expected that it will be equal to this as well, in which case it would act like about 7,000 cubic feet of ordinary coke-tower space, that is, twenty-five times its cubic capacity.

A very important advantage of the plate tower over the ordinary coke tower is the chemical indifference of the former to all the gases concerned, whereas the coke, as I have repeatedly proved, acts as a reducing agent upon the nitrous compounds and causes a loss of niter together with a gradual crumbling of the coke packing, while a plate tower lasts practically forever.

Another advantage of the plate column is its inconsiderable height, which not merely makes the pumping of the absorbing liquid much easier than with the ordinary coke tower, but in some cases admits of altogether dispensing with pumping. Thus, at the last-mentioned works the nitrous vitriol runs from the bottom of the plate tower serving as a Gay Lussac tower upon the top of the Glover tower, the former being placed at a convenient height for that purpose. This would have been impossible if the Gay Lussac had been an ordinary fifty or sixty foot coke tower. The matter might have been arranged just as easily in the opposite way.

The most important and interesting application of the plate column in the manufacture of sulphuric acid is that for diminishing chamber space. Every theory of the lead chamber process, worthy of such a name, supposes a constant interaction of the gases and the liquid portions floating in the chamber atmosphere in the shape of mist. Before that action is completed in the ordinary lead chambers, the ingredients must remain a very long time in contact with each other, and must quite gradually cool down, principally owing to the fact that the particles of the mist are separated by comparatively wide spaces and the action of the water or dilute acid on the nitroso-sulphuric acid is thereby greatly retarded. This drawback had been more or less clearly recognized by many previous inventors, beginning with Gossage, and many endeavors had been made to overcome it. But all these had either completely failed or at least effected their purpose so incompletely that they had been given up again. Thus all proposals for employing coke within the chambers for improving the mixture of the gases have necessarily broken down in consequence of the chemical action of the coke referred to above. Mechanical aspiration of the gases and re-injecting them into the chambers has also failed to improve matters to a great extent. Some little time ago the Thyss process attracted much attention. In this lead towers are interposed between the chambers, filled with lead sieves, but, as might have been foreseen, the lead sieves were soon stopped up by lead sulphate and quickly destroyed. The Delplace chamber is another attempt to produce a better mixture of the gases, but it can not attain this end at all completely or much better than any lead chamber of the old type.

By adapting my plate column to this special end, I believe, I have solved the problem more efficiently than any of my predecessors. I based my proposal on the fact, first securely established by myself, together with Dr. Naef, that the rate of formation of the acid within the first chamber of a set is very unequal. This was proved by very numerous observations and analyses, carried on at a Swiss works for several months, and afterwards confirmed at other places by a number of entirely independent observers. The curve of acid formation rises rapidly to a certain height, but about midways in a 100-foot chamber it flattens and turns nearly to the horizontal, so that the second half of the chamber performs very little work indeed. But as soon as the gases pass over into the second chamber, the formation of sulphuric acid is suddenly revived, thus proving that the contact of the gases and vapors with the end wall of the chamber, and their compression in the connecting pipe exerts a very favorable influence on their interaction. Most probably several conditions are at work here. The shock of the vapors against the solid surfaces, by which the sundry particles forming in their separation a fog or mist are brought together and are made to interact; further, the better mixture of the gases during their passage through the narrow connecting pipe, and lastly the cooling action of the end wall of the chamber, which cooling, according to the modern theories of the vitriol chamber process, is essential for the production of sulphuric acid from its components. Having thus, as I believed (and I think justly) recognised the causes of the favorable influence exerted by dividing the chamber space into several chambers, and of the revival of the reactions distinctly observable between the first and the second chamber, I proceeded to draw the consequence of these views, by immensely multiplying the conditions found to be conducive to the acid forming process. For this purpose I interposed, like Thyss, a kind of tower between the first and second chamber; but in lieu of simple lead sieves, which produce only an incomplete mixture, and which, as noticed before, are doomed to rapid destruction, I filled my tower with the plates I had previously devised for other purposes, consisting of chemically inattackable material, and by their geometrical design solving in the completest possible manner the problem of thoroughly dividing the gaseous current, and intimately mixing up its ingredients, together with innumerable shocks against solid surfaces. Even then two conditions for a proper chamber process would be absent, *viz.*, the supply of sufficient moisture, and the keeping down of the temperature below the optimum for the process; and these two conditions I supply simultaneously by feeding the towers with more or less dilute acid, whose evaporation yields both the water and the cooling action necessary for compelling the reaction to take the best possible course.

Theoretically there is no reason why such columns should not take the place of the lead chambers in their entirety. Long ago similar plans have been proposed, and quite recently a Mr. Barbier has again taken out a patent for a system, in which sulphuric acid is made without any lead chambers, in towers filled with earthenware cylinders, and fed with acids of different strength. In principle Barbier's plan does not at all differ from others which have been tried ten or twenty years ago, and have failed. Just that failure, I must confess, has discouraged me from proposing the entire abolition of the lead chambers to other people; but if I possessed a chemical factory of my own, I should certainly have tried that plan with my plate towers three years before Barbier took out his patent, which, of course, cannot be applied to that well-known principle, but only to his special kind of towers. For the present, at any rate, I do not venture to go beyond proposing to cut off the chambers at that point where all observations have proved the reactions to decrease rapidly in intensity, say, at a length of fifty feet, and to replace the remaining length of an ordinary chamber by a plate tower, repeating this arrangement with the second, and if needful with the third chamber of a set.

Up to this time no chamber system has been fitted up according to the plan just described, but in all cases the plate towers have been put between the existing chambers of an ordinary set, where they certainly perform a great deal of work, but evidently much less than could be done with a more perfect arrangement. I have therefore the right to consider the results hitherto obtained as a minimum; and looked at in this light, they are extremely encouraging, as far as I have been able to ascertain them, in which aim I have been much hampered by the unwillingness of many manufacturers to supply information for the benefit of their competitors. Thus for instance, the United Alkali Company, in England, has erected a plate tower, but I have not been able to extract any information as to the works at which that has been done, and for what purpose, let alone telling me anything about the results obtained therewith. In a few cases, fortunately, I have been favored with more information, and this I will now lay concisely before you.

At a Bohemian works, the first where this plan has been adopted, and where, consequently, the best conditions were not yet ascertained, the plate column has raised the quantity of pyrites burnt to an additional thirty per cent. Beyond this it is impossible to go in that place, as there is no more room in the kilns; but as the chamber following the plate column has no work whatever left to do, the acid making process having been completed in the column, the real increase of chamber capacity has been seventy per cent.

At a Dutch acid works there are two sets of chambers of exactly the same dimensions, namely one chamber of 64,400 cubic feet, and another of 19,950 cubic feet following this. In one of the two sets a plate column of ninety-six plates in all has been placed between the two chambers, and this set, I am informed, can be worked as if it held 22,500 cubic feet more than the other, so that each of the ninety-six plates would correspond to a space of 234 cubic feet, that is, more than two hundred times the room occupied by the tower itself.

At Valencia, in Spain, a plate column had been working for eighteen months, when I received the last news about it. That set was then burning forty-two per cent. pyrites in excess of what it had burnt before it had been provided with the column, whilst the consumption of niter had diminished from 2.2 down to 1.7 parts to 100 of fifty-two per cent. pyrites, which signifies a saving of nearly twenty-five per cent. of the niter. The yield of acid is excellent, and could hardly be better in that hot climate, where the first chamber in summer has a temperature up to 88° C. In the plate column it comes down to 28° or 50° C., and the gases go into the Gay Lussac with 40° C.

Looking at the very imperfect way in which the plate towers have been hitherto adapted to old sets of chambers, it is fair to hope that much better results than even those which I have been able to report would be obtained, if that principle were more thoroughly carried out. Even the present results show an immense superiority over Barbier's system, which, according to his own statement, requires one-sixteenth of the present chamber space, whereas my columns do from 100 to 250 times as much work as the same cube of chamber space. The reason of this great superiority evidently lies in the scientific construction of my plates, compared with the hap-hazard packing of other systems.

Allow me now to say a few words on the employment of plate towers for the condensation of hydrochloric acid. The very first tower which I designed was tried for this purpose, and the experience gained in that trial was utilized for a thorough reconstruction of the system. Since that time several plate towers have been supplied for the same purpose, and a very large one has been delivered just before my leaving Europe; but all information on the subject has been withheld from me, so that I cannot communicate anything on that subject beyond what was observed at the first trial, mentioned above, with the very imperfect kind of columns first used, at the celebrated Griesheim works, one of the most carefully managed alkali works in the world. According to the official report then furnished to me, the following results were obtained. Two superposed columns, of sixty cubic feet each, with a total plate surface of fifty-six feet (reckoning both top and bottom side of the plate) effected the condensation of ninety per cent. of the gases issuing from a salt cake pot, decomposing fourteen hundred weight of salt every four hours. The trial was continued for twenty-four hours in succession; the acid obtained varied from fifteen to twenty-two, and it averaged twenty degrees Baumé at 15° C. In England the smallest condensing space for such a quantity of salt would have been a coke tower of seven by seven by forty feet, holding about 200 cubic feet and possessing a coke surface of about

20,000 superficial feet. Compare that with my sixty cubic or fifty-eight superficial feet which did ninety per cent. of the work !

The coke tower certainly possesses one important advantage over its dwarf rival, the plate tower. The former holds a large stock of liquid, serving as a regulator for the unavoidable inequalities in the composition of the gases, whereas the latter possesses no such regulating stock. But this drawback can and ought to be counteracted by placing in front of the plate tower ten or fifteen stoneware receivers of the usual type, or a few large stone tanks, through which the acid condensed in the plate tower gradually flows onward, and issues from the last of the receivers at full strength; the gases are made to travel the opposite way, *viz.*, first through the receivers, and then through the plate tower. In this case the stock of acid contained in the receivers serves the same purpose as that contained in a coke tower.

Most of the 130 plate columns hitherto sold, serve for another purpose than those I have so far mentioned, *viz.*, for the recovery of nitric acid from the lower oxides of nitrogen by means of contact with air and water. I believe there is a general agreement on the point that no other existing arrangement for that purpose comes up to mine. In 1889, when the Jury of the International Exhibition at Paris listened to an explanation of the new methods for producing chlorine, based on the use of nitric acid, it was taken for granted on all sides that the problem of recovering nitric acid from nitrous fumes had been definitely solved by my apparatus. As far as the manufacture of commercial nitric acid is concerned, it is significant that both the rival inventors of new condensing systems for that acid, Mr. Guttmann and the Griesheim Company, much as they differ in every other way, employ the plate column at the end of their plant, for dealing with the nitrous vapors. In the just-mentioned case only a few per cent. of the total acid pass through the column ; but in all those cases where nitric acid is employed as an oxidizing agent, and where the lower nitrogen oxides, evolved in that process, had been formerly lost, or only quite incompletely recovered, the plate column now steps in as the coping stone of the edifice. Instances of this kind are the production of chlorine in any one of the processes doing this by the aid of nitric acid, with or without the intervention of manganese or other agents; the manufacture of arsenic acid from white arsenic, or of phthalic acid from naphthalene tetrachloride: the manufacture of iron mordant for dyeing purposes; that of blue copperas from metallic copper, and too many other cases to be enumerated here. Formerly, whenever the treatment of nitrous gases was at all attempted, this was done by a string of perhaps a hundred separate receivers, or by a coke tower, the latter being saddled with the great drawback already mentioned, *viz.*, its reducing action, in a case where the very opposite action is called for. In the March number of the *Journal of the Society of Chemical Industry*, Mr. Guttmann mentions the fact that at the works managed by him a small plate tower, ten feet high, performed the work formerly done by a coke tower forty-eight feet high, but much better; for the former made acid of 40°, the coke tower only such of 30°, and the plate tower actually satisfied the requirements of the Government Inspector, concerning the complete condensation of the nitrous gases, better than any other known apparatus.

In conclusion I beg to point out one more interesting use of plate columns. When gases have to be dried, by bringing them into contact with moderately concentrated sulphuric acid, which has generally been done by coke towers, the plate column is most evidently in its proper sphere, on account of its cleanliness, its chemical resistance to any attack, and the inconsiderable height to which the acid has to be pumped.

Many other uses of the plate column will no doubt suggest themselves to the industrial chemist; but I will bring my remarks to a close, and not detain you any longer with my invention.

DESIGN FOR WATER BATH.

By A. W. Nibelius.

THE following is a description of a water bath with constant water level, which I have found to be satisfactory in practical work:

Fig. 1 represents a longitudinal and Fig. 2 a transverse sec-