ON THE QUANTITATIVE DETERMINATION OF CONCREMENTS

II. Communication

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In our previous report the results were presented of comparative studies of methods for quantitative determination of calcium, magnesium, oxalate and phosphate ions in model solutions of concrements. The experimental data obtained indicate that the following methods may be considered the most appropriate:

1. Complexometric determination of Ca^{2+} with 0,002 M solution of complexon III with fluorexon — thymolphthalein as indicator.

2. Simultaneous complexometric determination of Ca^{2+} and Mg^{2+} a separate determination of Ca^{2+} and detection of the amount of Mg^{2+} through the existing difference.

3. Plumbometric determination of $C_2O_4^{2-}$ with 0,1 M solution of $Pb(NO_3)_2$.

4. Direct complexometric determination of PO_4^{3-} with 0,01 M solution of $MgSO_4$.

The purpose of the preliminary work was to evaluate contemporary methods for the determination of Ca^{2+}, Mg^{2+}, $C_2O_4^{2-}$ and PO_4^{3-} first on model solutions and then on native concrements. In the present work some results are reported of the quantitative study of concrements of renal or vesical origin. The materials are obtained at the Surgical Clinic and the Propedeutic Internal Clinic at the Higher Medical Institute in Varna. Data presented are compaired with data obtained by means of other methods.

Since the materials needed for the various determinations are already discussed in detail in our first communication, here only the analytic course and the results obtained will be presented.

Mode of work. The concrements, particularly if they are freshly formed, are carefully washed with cool water, dried about 30 minutes at 110°C and grinded in a mortar. From this material 0,5—2 g is measured and dissolved in an Erlenmeyer's flask of 100 ml in hydrochloric acid 1 : 1; it is then transferred to a mensure flask, water being added up to the mark. Qualitative investigations are performed with part of the solution for the composition of the concrement using the method described by Halman.

The preliminary qualitative analysis is followed by a quantitative processing of the material. In case the preliminary samples exhibit high contents of PO_4^{3-}, 25 ml of the examined solution is diluted eightfold in a mensure flask of 200 ml. In the diluted solution determination of Ca^{2+} and Mg^{2+} is feasible both after the classical sedimentation and volumetric methods, and after the complexometric methods offerred by us.

$C_2O_4^{2-}$ are determined on a sample from the initial solution, before the dilution.

When the preliminary qualitative analysis reveals that the concrement contains chiefly phosphates, a quantitative removal of PO_4^{3-} is performed by means of sedimentation in the form of ammonium phosphomolybdate after the method of Voy. The sediment is further dissolved in 8% ammonia and processed after the method described in communication I.

Samples of seven concrements are elaborated after the methods described in communication I. The results are shown on the table.

Table 1

Percentile content of Ca^{2+}, Mg^{2+}, $C_2O_4^{2-}$ and PO_4^{3-} determined in weight and volume by means of permanganometric and complexometric methods

Sample №	Weight of samplein g	Insoluble residue in HCl in %	g %			
			Ca^{2+} perm. comp.	Mg^{2+} perm. compl.	$C_2O_4^{2-}$ perm. comp.	PO_4^{3-} perm. comp.
1	0,7322	0,25	28,64 28,68	5,23 2,27	35,34	30,33 30,36
2	0.0445	—	18.55 18.60	2,18 2,22	49,47	29,78 29,83
3	0,0277	0,79	26,98 27,04	4,88 4,91	67,35	doesn't contc.
4	0,9188	4,68	50,66	1,15	33,20	2,94
5	0,7519	—	50,04	5,41	40,04	3,17
6	2,6354	1,02	16,14 16,60	6,07 6,05	doesn't cont.	41,52 41,74
7	0,4617	88,58	5,61 5,57	0,81 0,789	doesn't cont.	doesn't cont.

Discussion

The abovementioned data reveal a good correlation in the percentile content of ions, determined after various methods. The classical weight and permanganometric methods which are reliable, but slow and difficult to perform are utilized as a background for eliciting the advantages of the complexometric determinations, which are no less precise than the classical methods, but are more rapid and easily performed.

The quantitative content of samples 1 and 2 indicate that mixed concrements are concerned — phosphate-oxalate; sample No. 3 — oxalate concrement; sample No. 4 and 5 — mainly oxalate; sample No. 6 — phosphate concrement and sample No. 7 — typical uratic.

The insoluble residue indicates the presence of substances insoluble in hydrochloric acid. Here belong the residues of fats fatty acids, lipoids — cholesterin, steroid hormones, residues of cellular fragments, mucopolysaccharides, uric acid etc.

In some of the samples the amount of these substances does not exceed 1% and in such cases the data from the permanganometric and plumbometric determinations coincide. In a large percent of organic substances, as it may be expected, the permanganometric determination gives higher values, owing to an additional reduction of artefacts.

The percentile contents of sample No. 4 is quite different from 100 owing to the presence of ions, whose determination has not been the object of our work.

In sample No. 7 the insoluble residue amounts to about 89%. Obviously this is a typical uratic concrement.

The accumulation of further material for the analysis of concrements may facilitate the formation of a more precise view about the molecular structure of concrements.

Good coordination of data permits quantitative determination of concrements to be performed only after the complexometric methods offerred by us. Greater rapidity is thus achieved in the quantitative analysis of concrements. The quantitative analysis after the classical methods requires 2 and sometimes 3 work days for complete examination, whereas determination of the abovementioned ions requires 3—4 hours, including preliminary qualitative analysis.

For the quantitative determination of oxalate ions we consider the plumbometric method most appropriate since it renders the determination independent of the presence of organic substances. Otherwise a preliminary processing of concrements for the removal of the organic substances becomes necessary; for example fats and fatty acids are removed by rinsing with alcohol-ether solution, which complicates the analysis.

The methods offerred and tested by us may be successfully applied in clinical laboratory examinations for the solution of the problem about the most common ionic and molecular structure of concrements.

К ВОПРОСУ О КОЛИЧЕСТВЕННОМ ОПРЕДЕЛЕНИИ КОНКРЕМЕНТОВ

II-ое сообщение

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РЕЗЮМЕ

Сообщаются результаты определения Ca^{2+},Mg^{2+},$C_2O_4^{2-}$ и PO_4^{3-} в конкрементах. Данны о количественном составе получены на основании предварительно проверенных методов, использование которых, в целях анализа конкрементов, оказалось совсем успешным.

Точность примененных комплексометрических методов сравнена с точностью-классических. Результаты показывают хорошее совпадение, из чего следует, что предлагаемые комплексометрические методы вполне подходящи для быстрого и точного анализа конкрементов, содержащих вышеуказанные ионы.