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# **Evaluation of Mortar/Concrete Mix-Ratio Analysis Method**

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**EVALUATION OF MORTAR/CONCRETE  
MIX-RATIO ANALYSIS METHOD**

**By**

**Dr. Irshad Ahmed\***

**SYNOPSIS**

Need for mix-ratio analysis of a mortar or a concrete sample arises: to check how far specifications for the mortar/concrete have been adhered to by a contractor; to ensure uniform distribution of cement in a mix; to find out mix-ratio proportions in case of an unsound structure in order to assess the causes of its failure. Keeping in view flaws and demerits in laboratory and field methods for analysing cement content in mortar and concrete specimens, many experts developed new methods, especially for field purposes. The Irrigation Research Institute, Lahore has developed and published seven new rapid methods for mix-ratio analysis methods. In this paper an attempt has been made to evaluate all Mix-ratio analysis method.

## EVALUATION OF MORTAR/CONCRETE MIX - RATIO ANALYSIS METHODS

By  
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### Introduction:

Mortar is a mixture of inert siliceous material with an active ingredient, usually Portland cement, which, after being prepared into a workable plastic state with water, has the property of hardening into a compact mass.

The cement content being the only binding constituent, influences many of the important properties of mortar and concrete (when coarse aggregate is also used). Need for its estimation in freshly cast/hardened samples arises, in order to check how far specification of cement content have been adhered to. Its estimation also helps in assessing the uniformity of cement-sand dispersion throughout the mix. In addition, in an unsound structure, such an analysis is required in order to assess the causes of failure.

### Present Practice:

The mix ratio is generally assessed from mortar by indirect calculations which involve the elimination of lime and soluble silica. Concrete is also treated in a similar fashion and the process is duplicated on it after sieving and separating out the coarse aggregate. In this method it is desirable that the lime and soluble silica contents in the constructions (cement sand and aggregate) of the material are known. The method is restricted whenever an aggregate that liberates soluble silica under test conditions is used in concrete.

Chemical methods<sup>1</sup> commonly used are not only laborious and time consuming, but also require a well-equipped laboratory and services of a well trained chemist. Realising the need for development of simple, rapid cement estimation methods; a number of methods are put forth by various workers. These methods with their evaluations are briefly given below:

#### i) Conductivity Method<sup>2</sup>

This is a computation method, applicable only to unset cement sand mixtures, and is based on determining the conductivity of the water in which a known quantity of unset cement-sand mixture has been vigorously shaken. Cement, coming in contact with water, releases free lime, the concentration of which is directly proportional to the percentage of cement present in the mixture. The conductivity of the resulting solution is measured with a calibrated electric device.

This method suffers certain backs such as those listed below:

- i) This is meant only for freshly prepared mixes.
- ii) This gives inconsistent results owing to the presence of variable salts in mixing water at different sites.
- iii) Slight impurities present in the mixing water and setting of cement during shaking affect

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the results obtained by the conductivity method.

ii) **Adsorption Method<sup>2</sup>**

This method is based upon the differential adsorption characteristics of cement and sand particles. The percentage adsorption, being directly proportional to the cement content, increases as the concentration of cement increases in the mixture. The mortar (10 g) passing 40 mesh is shaken with potassium permanganate ( $\text{KMnO}_4$ ) (40 ml of 0.2 N) solution for half an hour and filtered. The quantity of  $\text{KMnO}_4$  adsorbed is determined by back titrating with standard oxalic acid solution ( $\text{C}_2\text{H}_2\text{O}_4 - 2\text{H}_2\text{O}$ ). The adsorbed  $\text{KMnO}_4$  determines the cement content of the sample. For easy interpretation of the results, a curve of cement content versus adsorbed  $\text{KMnO}_4$  is available. The adsorption capacity of the clay complex interferes with the results obtained by the adsorption method, so consistent results are seldom obtained in practice by this method.

iii) **Potassium Permanganate-Titration Method<sup>3</sup>**

This resembles the method specified by the ASTM designation C85-54. The mortar (1 g) is digested with hydrochloric acid (HCl) and the solution thus obtained is filtered and washed. It is then neutralised with liquid ammonia. The precipitates of mixed oxides are dissolved by glacial acetic acid. A saturated solution of ammonium oxalate is then added to precipitate calcium oxalate. The precipitate is washed, filtered and subsequently treated with sulphuric acid, and after titrating with standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution, the calcium content is estimated. The calibration curve of cement content versus calcium content is used.

The method cannot be considered very simple and rapid as it contains many chemical steps, which can be followed accurately by an experienced chemist.

iv) **Selective Solution Method<sup>4</sup>**

The method, introduced by Tabikh and his co-workers, involves washing of dried and crushed concrete/mortar sample with a methanolic solution of maleic acid. The hydrated and unhydrated calcium silicates and the hydrated aluminates and ferrites are selectively dissolved by the washing, leaving the unattacked aggregates plus the unhydrated aluminates and ferrites as residue to be separated by filtration. From the weights of the residue, the water combined in hydrated cement, the free water, and the bulk specific gravity of the concrete sample, the cement content is calculated and expressed in terms of percentage.

The above method is expensive as it uses large quantities of costly chemicals and is also time consuming.

**Rebound Hammer Method:<sup>5-9</sup>**

Rebound Hammer Method is used for measuring surface hardness of hardened mortar and concrete. It is based upon the height of rebound of a standard steel rod when dropped on a hardened mortar or concrete from a given height. It, thus, gives the strength of cement and indirectly cement content.

This method is not dependable as it is very approximate. In concrete, this form of test gives very variable results, particularly when larger pieces of aggregate are close to the surface at the point at which the test is done.

v) **Nuclear Techniques<sup>10-13</sup>**

Covault and Possey's neutron activation analysis method, to assess cement content, is based on determining the amount of cement in a radioactive concrete sample by counting the radioactivity and determining the cement content from the cement content versus rate curve. The method requires costly radiation and counting equipment. It cannot be used with aggregate containing

appreciable amounts of calcium. Feasibility of other nuclear techniques such as activation analysis, stable tracer analysis, natural radioactivity measurement, isotope dilution for determining the cement in concrete, have also been investigated by Iddigns and his co-workers. These techniques are, however, either applicable only under ideal conditions or are not economical. The safety of personnel against potential radiation hazards must also be ensured when using these techniques and this imposes an extra cost on the analysis.

In view of the demerits discussed in the rapid Cement Estimation Methods, the research work in this field was initiated by the author of this paper in the Irrigation Research Institute, Lahore. During last 20 years' of research in the institute, many existing methods were improved, many new ones were developed, and thus published seven papers on improved and newly developed cement estimation methods, mentioned below:

- i) Potassium Permanganate Capsule Method
- ii) Improved Conductivity Method
- iii) Average Conductivity Method
- iv) Residual Conductivity Method
- v) Reduced Volume Method
- vi) Improved Reduced Volume Method
- vii) Organo Volume Method

Most of the above methods were developed by the author while working as Principal Investigator on a PL-480 funded Lining Project<sup>14</sup>, initiated by the three organisations: the United States Department of Agriculture, the Agricultural Research Council Government of Pakistan and the Irrigation Research Institute, Lahore. The Institute is still active in this field and two or three more publications are expected on this subject.

The rapid cement estimation methods, developed by the author in the Institute are briefly discussed below:

**i) Potassium Permanganate Capsule Method<sup>15</sup>**

This method is similar to that developed by Dewan. The process of adding hydrochloric acid to 1 gm powdered sample, filtering, washing, adding, aqueous ammonia, precipitation of mixed oxides as hydroxide and filtering the resulting solution are carried out in a similar manner to the said method. The last two operations of adding ammonium oxalate and sulphuric acid are also performed. However instead of next titrating with  $\text{KMnO}_4$  solution, glass capsules filled with a known quantity of solid  $\text{KMnO}_4$  are added one by one until the colour of the solution changes to pink. The number of capsule units added directly determines the percentage of lime present.

**ii) Improved Conductivity Method<sup>16</sup>**

The conductivity method, developed by Chadda, does not give consistent readings, owing to setting of cement during shaking of mortar sample.

In the improved conductivity method the problem of setting of cement during shaking process with distilled water for conductivity measurement, has successfully been solved by shaking mortar in 0.5 percent sugar solution which appreciably delays setting of mortar and thus helps in getting consistent conductivity readings. The improved conductivity method can, now, be applied even to quick setting cements. Moreover, washing of conductivity bottles and conductivity tubes has been made convenient by delaying the setting time of cement.

iii) **Average Conductivity Method**<sup>17</sup>

This method involves only the determination of average conductivity of mortar in the range of 10 to 50 per cent cement contents mortar and that of unknown samples. The method is rapid, fairly reliable and suitable for field work. The following mathematical relation has also been derived to facilitate calculations:

$$P_U = P_S - \left( \frac{C_S - C_P}{0.025 (C_{50} - C_{10})} \right)$$

Where

- $P_U$  = Percentage of cement used
- $P_S$  = Specified percentage of Cement for a mix.
- $C_S$  = Conductivity of a mix of the specified ratio.
- $C_P$  = Conductivity of the mortar sample. (i.e. prepared by contractor).
- $C_{50}$  = Conductivity of a self-made 50 percent cement content mortar.
- $C_{10}$  = Conductivity of a self-made 10 percent cement content mortar.

iv) **Residual Conductivity Method**<sup>18</sup>

This method involves conductivity measurements of the known and unknown mortar samples. Difference between conductivity of known and unknown samples of mortar ( $C_S - C_P$ ) is termed as 'Residual Conductivity' ( $C_r$ ) and its sign ( $\pm$ ) not only gives qualitatively whether a mortar is richer or leaner than the specified mix-ratio but it also helps in finding out quantitatively the ratio of an unknown sample. The method is rapid, fairly accurate and highly helpful in checking uniform distribution of cement in a mix. The mathematical relation developed for the method is as follows:

$$C_S - C_P = \pm C_r$$

Three possibilities of a mix sample under test are:

- a) If  $C_r = 0$ , then prepared mix ratio is equal to the specified one.
- b) If  $C_r = +$ , then the prepared mix ratio is leaner than the specified one.
- c) If  $C_r = -$ , then the prepared mix ratio is richer than the specified one.

v) **Reduced Volume Method**<sup>19</sup>

All the present chemical methods are based on lime and silica determinations. Occasionally, where lime has been added in excess of that usually present in a cement, the soluble silica content is determined instead. Complications also ensue in the case of siliceous and magnesium limestones. Assumptions must be made when the original cement – sand and aggregate are not available at the time of test.

These difficulties can be countered to some extent by working out the cement content on the basis of constituents other than silica or lime. The method described in this paper is based on determining the volume of the inert matter present in a sample of mortar or concrete. The method is perfected with a volume measuring tube (VMT) adopted by Puri<sup>20</sup> while performing size distribution studies on samples of silts and sands.

**Principle:**

When a sample is intermixed and shaken with an acid (HCL), the reactants are dissolved out, leaving behind the inert matter and soluble silica content of the sample. The residue, when subsequently made to react with an alkali ( $Na_2CO_3$ ) leaves behind the inert matter that comes mostly

from the aggregate. Measurement of this matter can provide a quantitative indication of the cement content in the mortar or concrete.

#### Test Procedure:

The pulverised and carefully homogenised mortar test sample (2 g) was placed in a beaker and treated with distilled water (20 ml) and hydrochloric acid (10 ml; specific gravity 1.16). The contents of the beaker were warmed gently for 5 to 10 min and after being allowed to settle for about 5 min, were filtered through a Whatman No. 41 filter paper. The residue washed with cold water, was heated below boiling point for about 15 min with sodium carbonate (30 ml of 5 percent solution), and the filtered again. The inert matter remaining on the filter paper was washed with a few drops of HCl (1:9) and finally with hot water. It was then transferred in the wet state into the volume measuring tube (VMT). The VMT was tapped gently at intervals on rubber paddings for about 10 min to record the volume of the insoluble solid mass. Blank determinations with the same quantities (2 g each) of cement and sand samples ground to pass completely through the 52 mesh sieve, were also run simultaneously, under similar conditions. The average of two observations was considered sufficient to calculate the cement content in a sample. The VMT used for recording the observations is shown in Fig. 1(a) and 1(b).

To confirm the accuracy and validity of the above technique, tests were repeated in a similar pattern except that, instead of reading the volume of inert matter in the VMT, it was transferred into a pre-weighed crucible and dried in an electric oven at 110°C for about 24 h. The weight of the dried residue was recorded for subsequent calculations.

The observed volumes of the residue for mortar test samples and for the constituting materials (cement and sand) were put in the formula:

$$C/S = \frac{V_s - V_m + V_c}{V_m - V_c}$$

where

- $V_s$  = VMT volume for Sand (c.c.)  
 $V_m$  = " " " C : S mortar (c.c.)  
 $V_c$  = " " " Cement (c.c.)

#### vi) Improved 'Reduce Volume' Method<sup>21</sup>

With the reduced volume method, two difficulties have been observed.

- 1) Accurate readings of the insoluble residue (R) column is often difficult with the naked eye.
- 2) To keep the volume measuring tube (VMT) verticle while taking readings is often difficult with the usual VMT stand.

These drawbacks prompted some refinements to enable more accurate readings to be made, as follows.

#### Apparatus:

The equipment, shown in Fig. 2, developed for the 'improved reduced volume method', consists primarily of a VM tube (ref. 20), as used in the original reduced volume method, with a more accurate arrangement for taking readings.

The apparatus consists of a specially designed plastic mounting for holding the VM tube in a vertical position. This mounting is fixed to a wooden frame which rests on a metallic platform with leveling screws, thus holding the VM tube in the vertical position, as indicated by a bubble tube fixed on the board.

Readings are taken with the help of two vernier scales secured parallel to each other on the wooden frame. A horizontal wire, which can be moved across the scales through a rack and pinion, enables accurate reading to be made of the upper and lower edges of the IR column. A mirror placed behind the horizontal wire to avoid parallax error.

The reading of the IR column in the VM tube can be taken up to 0.01 cm with the vernier scale.

#### Discussion:

The validity of the RV method was examined by preparing the test specimens under controlled laboratory conditions. The concentration of cement in these specimens ranged from 1 : 1 to 1 : 10 cement, sand. In order to observe the effect of the curing periods on the results obtained by this method a number of samples with the same cement content were prepared and cured for three, seven and 28 days so that similar samples of each concentration for the above mentioned period were available in duplicate for testing. The volume of the inert matter measured in the VMP after the proposed operations and the C.S. ratio calculated on the basis of this volume along with the actual C.S. ratio of each sample, is given in Table. 1. As can be seen from this table, the cement content determined by the RV method is generally in agreement with the actual sand cement content in almost all the specimens.

As a counter-check on the results obtained by the RV method, duplicate samples of mortar and concrete were also analysed by determining the weight of insoluble residue left after the treatment of both HCl and  $\text{Na}_2\text{CO}_3$ . The cement contents pertaining to each specimen cured for different periods are given in Table. 2.

For each application of the method and rapid interpretation of the results, a simple mathematical relation has been derived. Reference curves of the cement content and mix ratios versus VMT volume/weight of the insoluble residue have also been drawn and are given in Figs. 3 & 4 thus the mix ratios or the cement content of a sample under examination can be directly interpreted provided the VMT value is known. The determined VMT volumes versus the equivalent amounts of cement actually present/practically observed have also been tabulised (Table 3) as a further aid in the rapid interpretation of the results.

As a prerequisite of the method, the original sand used in the preparation of the mortar should be available at the time of analysis in order to accurately assess the mix ratio. Since such samples are not usually available in practice, the VMT volumes of the inert matter present in different varieties of local sands were also determined; the results are given in Table. 4. This table shows that the presence or absence of the original ingredients of a mortar are not likely to produce marked effects on the mix ratios of the sample.

Actual mix ratios of the concrete sample cured for specific time periods and the determined on RV basis, mix ratios are given in Table. 5. These results clearly indicate that the method works equally well for concrete samples.

#### Relative Advantage of the Method:

- 1) The method is based on determining the inert matter of a mortar or concrete sample and



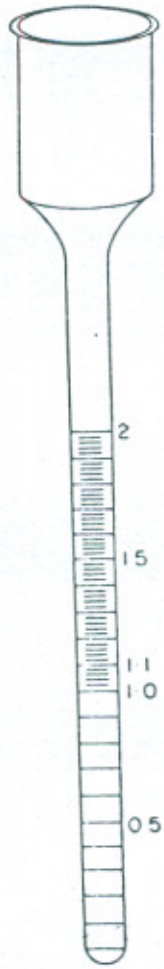


Fig. 1(a). *Volume measuring tube.*

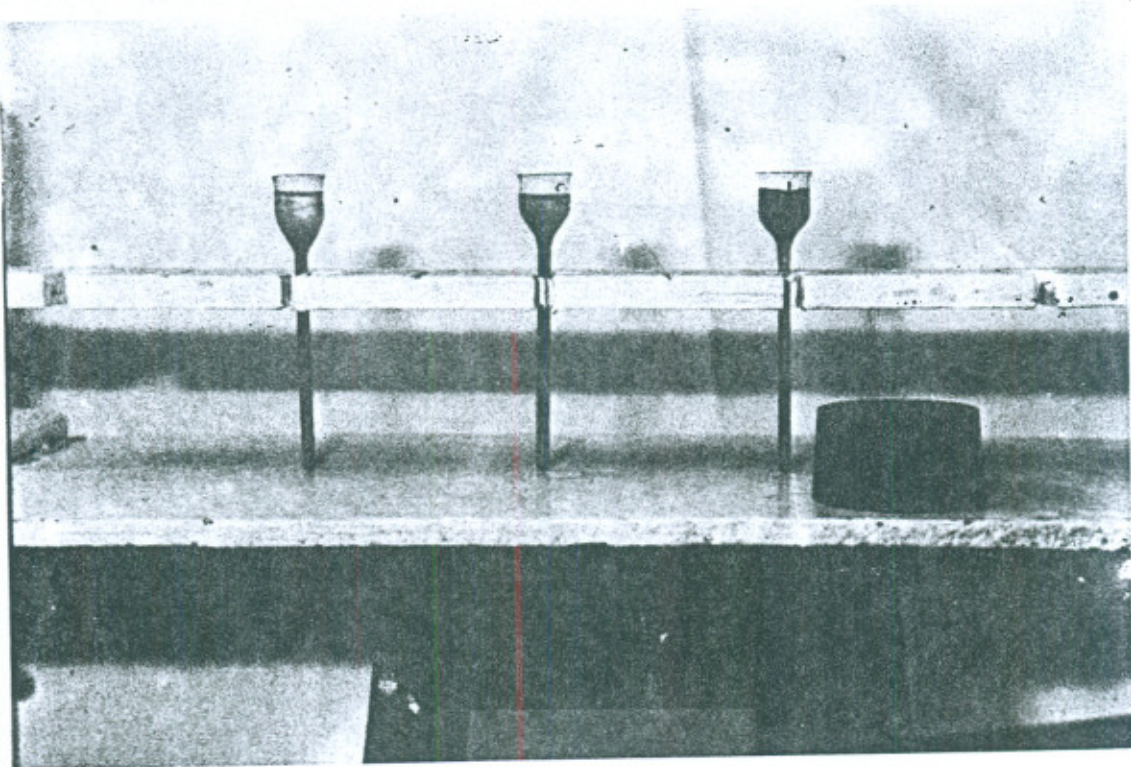


Fig 1 (b) VMT in Operation

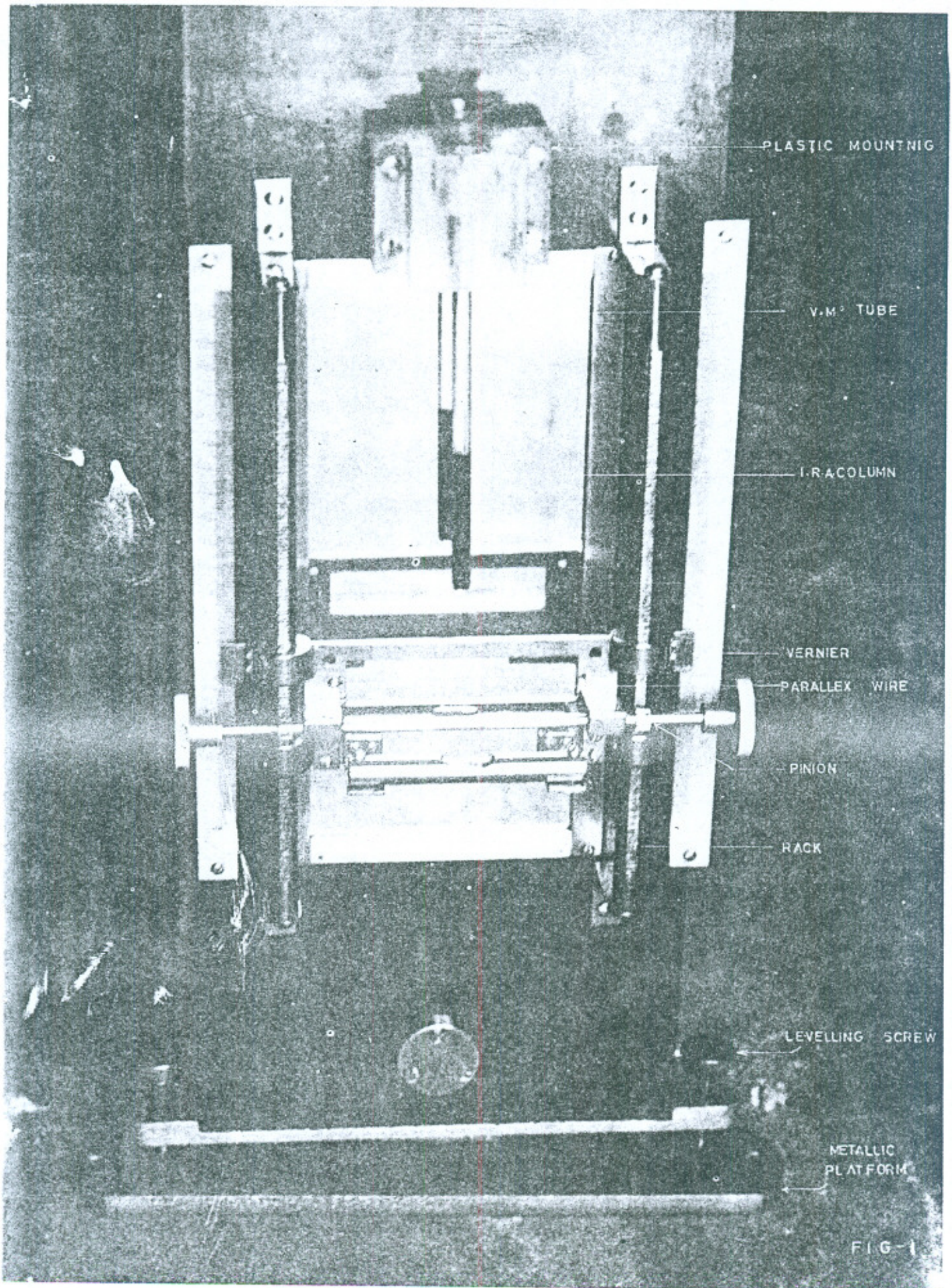


Fig. 2 Equipment for Improved Reduced volume method

Fig. 3

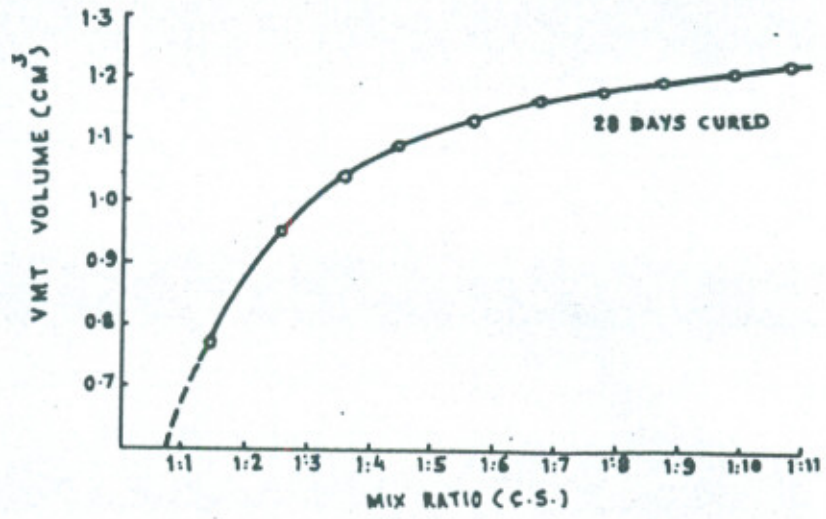
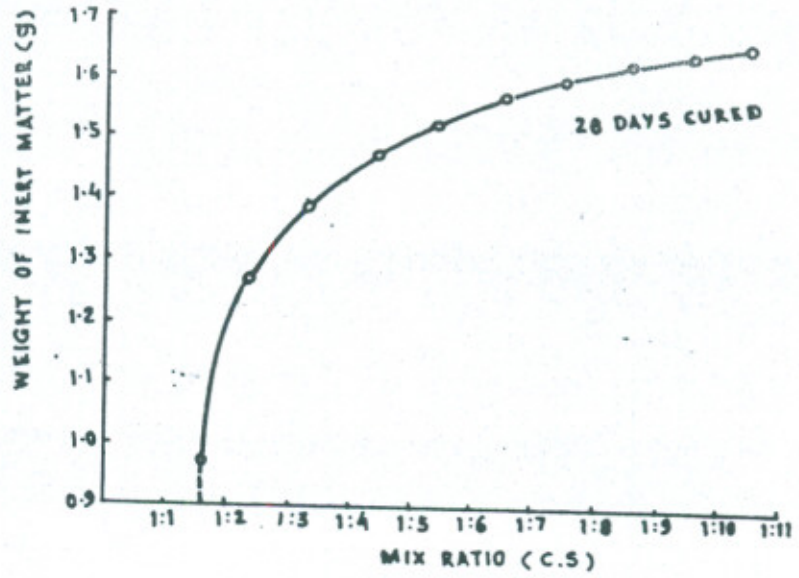


Fig. 4



thus the question of interference from the limestones, siliceous sand-stones and dolomite types of coarse aggregate, usually encountered in other methods, does not arise.

2) It does not require the elimination of radicals (for example the mixed oxides), which otherwise must always be removed by precipitation, etc. before the sample is analysed.

3) Standard solutions of the reactants are not used, with consequent saving of labour and time.

4) The standard graphs avoid the need for computation.

5) A mathematical relation directly interprets the results.

6) The method can be applied with confidence both in the laboratory and in the field.

7) Availability of the constituent materials is not likely to pose a serious problem in determining the mix ratio.

8) It is a simple method and does not involve the normal weighing and chemical operation. A field engineer with a kit containing the acid, alkali solution, filter papers, beakers, funnels and the VMT would be able to assess the mix ratio being used and thus the uniformity of the mix throughout the construction batch would be effectively maintained.

#### **Organo Volume Method<sup>22</sup>:**

Tabikh and Clemena's selective solution mortar analysis for determining mix ratio involves several time-consuming steps. In organo volume method, developed by the author, an attempt has been made to modify and further simplify it by eliminating many chemical and weighing operations, with the use of a volume measuring tube, Methanol has been substituted by methylated spirit to fairly reduce the cost factor. Appreciably reliable results are obtained with this method, provided the suggested precautionary measures are properly applied. The method is quite simple to be adopted easily, by an average worker without any difficulty, for rapid determination of cement content in mortar and concrete.

The above mentioned simple economical, rapid cement estimation methods, developed for different situations, may contribute to improve the quality of construction work.

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Table 1 VMT volumes and equivalent actual/observed mix ratios of test specimens  
 (i) VMT volume of Sand\* used in test specimens (C:C) =  $V_s = 1.34$

Test specimen No.	Actual MR + (C:S)	Dry unmixed				Cured			
		VMT $\ddagger$ volume	C:S (ii)	Three days		Seven days		28 days	
				i	ii	i	ii	i	ii
AB	1:1	0.63	1:0.89	0.71	1:1.13	0.76	1:1.31	0.77	1:1.35
CD	1:2	0.88	1:1.91	0.92	1:2.19	0.94	1:2.35	0.95	1:2.44
EF	1:3	1.01	1:3.06	1.02	1:3.19	1.03	1:3.32	1.04:3.46	
GH	1:4	1.07	1:3.96	1.08	1:4.15	1.09	1:4.36	1.09	1:4.36
IJ	1:5	1.12	1:5.09	1.125	1:5.23	1.13	1:5.38	1.135	1:5.54
KL	1:6	1.15	1:6.05	1.155	1:6.24	1.16	1:6.44	1.165	1:6.66
MN	1:7	1.175	1:7.12	1.18	1:7.38	1.18	1:7.38	1.185	1:7.65
OP	1:8	1.19	1:7.93	1.195	1:8.24	1.195	1:8.24	1.20	1:8.57
QR	1:9	1.20	1:8.57	1.21	1:9.31	1.21	1:9.31	1.215	1:9.72
ST	1:10	1.215	1:9.72	1.22	1:10.17	1.22	1:10.17	1.225	1:10.65

\* Ground to pass a 52 mesh BS sieve.

† Mix ratio.

‡ Average of two determinations.

Table 2 Weight of inert matter and equivalent actual/observed mix ratios of test specimens  
 (i) Weight of inert matter of Sand\* used in test specimen (g) =  $W_s = 1.810$

Test specimen No.	Actual MR C:S by wt.	Dry unmixed		Cured					
		Wt† of inert matter (g) (i)	C:S (ii)	Three days		Seven days		28 days	
				i	ii	i	ii	i	ii
AB	1:1	0.926	1:1.05	0.945	1:1.11	0.960	1:1.13	0.973	1:1.16
CD	1:2	1.250	1:2.23	1.253	1:2.25	1.260	1:2.29	1.270	1:2.35
EF	1:3	1.372	1:3.13	1.374	1:3.15	1.382	1:3.23	1.386	1:3.27
GH	1:4	1.450	1:4.03	1.460	1:4.17	1.470	1:4.32	1.470	1:4.32
IJ	1:5	1.515	1:5.14	1.515	1:5.14	1.520	1:5.24	1.525	1:5.35
KL	1:6	1.556	1:6.13	1.560	1:6.24	1.565	1:6.38	1.568	1:6.47
MN	1:7	1.580	1:6.87	1.585	1:7.04	1.595	1:7.42	1.596	1:7.46
OP	1:8	1.608	1:7.96	1.613	1:8.19	1.617	1:8.38	1.620	1:8.53
QR	1:9	1.624	1:8.73	1.631	1:9.11	1.635	1:9.34	1.638	1:9.52
ST	1:10	1.641	1:9.71	1.643	1:9.84	1.650	1:10.31	1.652	1:10.46

\* Ground to pass a 52 mesh BS sieve.

† Average of two determinations.



Table 3 VMT volumes and equivalent amounts of actual observed cement contents  
(28 days cured specimens)

Test specimen No.	Actual cement content (%)	Dry unmixcd		Cured					
		VMT vol. C:C (i)	Cement content % (ii)	Three days		Seven days		28 days	
				(i)	(ii)	(i)	(ii)	(i)	(ii)
AB	50.0	0.63	52.9	0.71	46.9	0.76	43.3	0.77	42.6
CD	33.3	0.88	34.4	0.92	31.3	0.94	29.9	0.95	29.1
EF	25.0	1.01	24.6	1.02	23.9	1.03	23.1	1.05	21.6
GH	20.0	1.07	20.2	1.08	19.4	1.09	18.7	1.09	18.7
IJ	16.7	1.12	16.4	1.125	16.1	1.13	15.7	1.135	15.3
KL	14.3	1.15	14.2	1.155	13.8	1.16	13.4	1.165	13.1
MN	12.5	1.175	12.3	1.18	11.9	1.18	11.9	1.185	11.6
OP	11.1	1.19	11.2	1.195	10.8	1.195	10.8	1.20	10.4
QR	10.0	1.20	10.4	1.21	9.7	1.21	9.7	1.215	9.3
ST	9.1	1.215	9.3	1.22	8.95	1.22	8.95	1.225	8.6

Table 4 VMT volumes of local sands\*

Source	VMT volume† (C:C)	Variation limit (%)	
		When Ravi sand is assumed as standard i.e. 1.34	When Av. of VMT volumes of local sands is assumed as standard, i.e. 1.38
Chenab	1.37	+2.24	-0.73
Harrow	1.44	+7.46	+4.36
Karachi (Thana Bola Khan)	1.34	-	-2.9
Lawrence	1.38	+3.0	-
Mari	1.40	+4.48	+1.46
Ravi‡	1.34	-	-2.9

\* All ground to pass a 52 mesh BS sieve.

† Av. of two determinations.

‡ Used in the test specimens.

Table 5 VMT volumes and equivalent actual/observed mix ratios of concrete test specimens  
(i) VMT volume of sand\* used (C:C) =  $V_s = 1.34$

Test specimen No.	Weight of specimen (g)			Actual MR C:S:G	VMT volumes and observed MRS						
	Curing period (days)	Total	Gravel		Mortar (C:S)	Three days VMT† volume (i)	Three days C:S:G (ii)	Seven days (i) (ii)		28 days (i) (ii)	
BD	3	1158	665	493	1:1.50:3	0.78	1:1.39:3.22	0.83	1:1.62:3.4	0.88	1:1.91:3.1
	7	1176	663	513							
	28	794	409	385							
FU	3	1317	707	610	1:2:4	0.93	1:2.27:3.8	0.92	1:2.19:4.2	0.85	1:1.73:4.7
	7	1448	823	625							
	28	1509	954	555							
OK	3	1265	750	515	1:3:6	1.03	1:3.32:6.3	1.035	1:3.40:6.5	1.02	1:3.19:6.8
	7	1298	774	524							
	28	1537	952	585							

\* Ground to pass a 52 mesh BS sieve.

† Average of two determinations.