TC 178-TMC: "Testing and Modelling Chloride Penetration in Concrete"

Recommendations

METHODS FOR OBTAINING DUST SAMPLES BY MEANS OF GRINDING CONCRETE IN ORDER TO DETERMINE THE CHLORIDE CONCENTRATION PROFILE

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The text presented hereafter is a draft for general consideration. Comments should be sent to the TC Chairlady: Dr. Carmen Andrade, Instituto de Ciencias de la Construcción "Eduardo Torroja", Serrano Galvache nº 4 – Apartado 19.002, 28033 Madrid, Spain
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Content

1 SCOPE

This RILEM Technical Recommendation intends to give a general description of methods of sampling for obtaining chloride concentration profiles in concrete, applicable both for laboratory cast concrete specimens, for concrete cores taken from structures and for testing on site. These sampling procedures may be applied for obtaining concentration profiles of any other chemical species present in concrete.

2 SIGNIFICANCE AND USE

The chloride concentration profile in concrete from the exposed surface inwards is a valuable tool for assessing the risk of corrosion of reinforcement steel in concrete structures exposed to marine or de-icing salt environments. From the knowledge of the Cl^- profile of a structure at a given age, one can get some qualitative information about the rate of ingress of such ions into the structure, and then predictions about the reinforcement corrosion onset time can be formulated. These assessments are highly improved when several Cl^- profiles are obtained at different times for the same structure. Furthermore several test methods for obtaining Cl^- transport parameters in non-steady-state conditions are based on fitting the experimental Cl^- content profile to a particular equation. These transport parameters can be used as input parameters of mathematical models intended to predict the corrosion initiation time. The first step for obtaining the profile is sampling, that may be done on-site or at laboratory on cores previously taken from the structure or concrete specimen.

3 DEFINITIONS

The concentration profile of a chemical species in concrete can be defined as the spatial distribution of this species in concrete. This distribution is a function of the exposure conditions and time [1]. The chloride profile in the reinforcement cover zone of concrete is a set of data pairs (depth from surface-concentration) obtained by chemical analysis of chloride on representative samples taken from the concrete specimen or structure. Usually the profile is presented in tabular or graphical form. The depth from surface of each sample is expressed in length units, and the units of the concentrations must be clearly established, i.e. % chloride referred to concrete mass. Concrete contains free chloride in its pore solution, bound chloride (chemically or physically bound or trapped by solid phases of concrete). This suggests that several chloride profiles can be considered or obtained, (total, free or bound chloride), depending on the fraction considered, or on the analytical method used for determining the chloride content (acid soluble fraction, water soluble fraction, etc.) [2].

Regarding the sampling procedures and in order to avoid confusions the following definitions are proposed:

A) Dry drilling method: Sampling by drilling with a rotary hammer and a masonry bit, working directly on the surface of a concrete specimen or real structure. This procedure allows obtaining powdered samples corresponding habitually to depth steps of about 5 mm.
B) Grinding method: Sampling by grinding the surface of a concrete specimen or real structure.

C) Cutting and crushing method: Sampling by saw cutting concrete slices of adequate thickness, (usually not less than 10 mm), from a specimen or core, and posterior crushing of the slices manually (mortar and pestle) or mechanically (ball or ring mills), to get the powdered sample.

4 GENERAL CONSIDERATIONS OF THE SAMPLING PROCEDURES

When sampling one of the most important needs is getting representative samples. The size of the samples must be large enough to fulfil the sample requirement of the analytical chemistry procedure; and to avoid errors due to local heterogeneity, for instance between cement paste and aggregate zones, whose contributions to the chloride content may be quite different. For this reason the minimum amount of sample is related normally with the maximum size of aggregates [1]. Care must be taken also in gaining the test samples from the concrete element in order not to affect the chloride content [1]. The modifications to the original Cl^- content of the samples can be due to contamination with portions of material coming from another sampling point [3]; to partial washing of the soluble chlorides if water is used as a lubricant during drilling or cutting the samples; or to redistribution of Cl^- ions during the sampling due to moisture transport. This latter situation may be produced for the case of cores taken from a structure, to be cut and crushed later at laboratory, if they are not adequately protected from drying.

If a realistic concentration profile must be established from experimental analyses, samples must be taken from the concrete element at different and known depths from the surface, i.e. each concrete sample for the Cl^- profile must be associated to a particular depth [4]. A high spatial resolution is required if especial effects are to be detected, such as surface wash-out, or if transport parameters are to be derived from the profiles [1]. The determination of Cl^- profiles for high performance impervious concretes, with a high resistance to Cl^- penetration, may require also the selection of a sampling procedure with a high spatial resolution.

Other factors which should be considered when selecting the sampling procedure are the costs and time consumptions, associated with the sampling and analysis of the samples; and the damages to the appearance and integrity of the structure, which may be important for instance when performing extensive coring. The more accurate are the methods of sampling, the higher are the costs, time consumptions and eventual damage, since the high resolution procedures usually need high diameter cores or sampling areas. This calls for a compromise between the interest of sampling at several different points of a structure, to get representative information of the behaviour of the whole structure, and the need of obtaining detailed profiles, in order to increase the reliability of the derived transport parameters.

In the case of sampling on site, a previous step will consist in the selection of the areas where the chloride profiles will be determined or cores drilled for so. This selection is out of the scope of present Recommendation as it will depend on the purpose of the inspection or particular test.

5 METHODS OF SAMPLING

5.1 Dry drilling method

This procedure consists in collecting the concrete dust obtained when drilling small diameter holes on the concrete surface, using a rotary hammer and a masonry bit, see Fig. 1. The drilling is performed in steps of selected thickness. The number of steps in the profile and their thickness must be decided taking into account the estimated depth reached by the penetrating chloride front and the desired spatial resolution. Nevertheless, a minimum of 5-8 steps are needed for obtaining a detailed enough profile, and the recommended minimum thickness of the steps is 5 mm, although it is possible to obtain samples at steps of 2.5 mm.

The dry drilling method may be considered as a simple, cheap and less destructive sampling method, as compared for instance with the grinding method, since it does not require taking cores of large diameter. All these characteristics may allow considering the method as more suitable for performing extensive sampling campaigns. On the other hand the dry drilling is usually a sampling method of low spatial resolution, due to the minimum thickness of the drilling steps (about 5 mm), as compared with the grinding procedure.

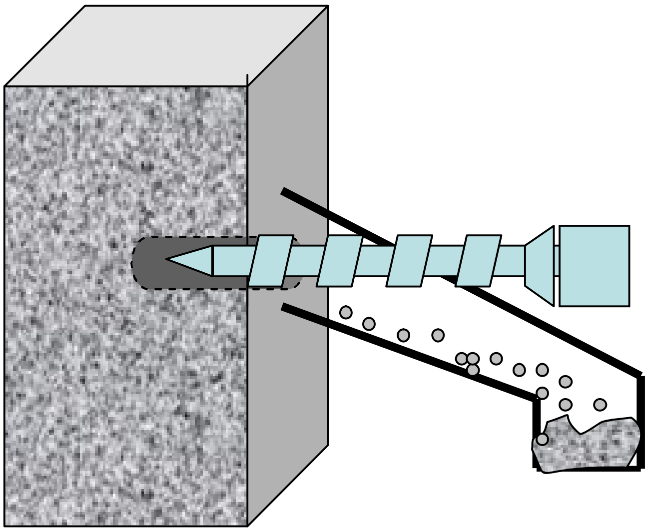

Figure 1. Disposition of drilling machine for sampling concrete dust.

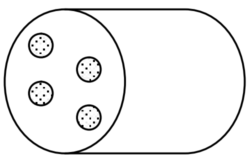

Figure 2. Drilling holes for collecting dust.

The collection of concrete dust during dry drilling is performed with specific dust collectors attached to the concrete surface, see Fig. 1, or more conveniently by using a vacuum attachment that allows sampling from surfaces in all directions, even upwards from a horizontal surface. The size of the concrete dust samples must be large enough to guarantee a representative profile and to enable an accurate determination of the chloride content by the corresponding chemical procedure (minimum 5 g of dust for each sample). The amount of dust obtained depends on the diameter of the holes, hence on the diameter of the bit, and on the thickness of the drilling steps. A minimum bit diameter of 18-20 mm is recommended to avoid excessive contributions from large aggregates; although bits of lower diameter, (10-12 mm), could be used for laboratory tests on mortar or cement paste specimens. Another useful means for obtaining more representative profiles, or for increasing the amount of concrete dust obtained for each sample, is to perform several drillings in the surroundings of each point to be studied in a structure, mixing the dust samples corresponding to each hole for a given depth step, see Fig. 2. The minimum number of drilled holes recommended can be established in function of the bit diameter and the maximum aggregate size of the concrete, for a certain thickness of the drilling steps [5].

Table 1. Recommended number of drill holes for collecting concrete dust samples, in function of the hole diameter and the maximum size aggregate, for a 10 mm thickness of the drilling steps [5].

Max. aggregate size (mm)	Number of drill holes with diameters			
	20 mm	26 mm	32 mm	40 mm
8	1	1	1	1
16	2	1	1	1
32	5	3	2	1

The risk of modifying the chloride content of the concrete dust samples obtained is low since the drilling is performed in dry conditions; provided that a proper mechanical fixation of the drilling machine is ensured for obtaining a perfectly perpendicular drilling to surface [3]. Another possibility to decrease the risk of contaminating the concrete dust samples due to lateral contact of the bit with the wall of the bore hole at lower depths, is the use of two bits of slightly different diameters, for instance 18 and 22 mm. After obtaining each sample with the lower diameter bit, the bore hole can be widened by passing the higher diameter bit through the same depth step. Thus, the next sample can be taken without lateral contact of the bit (lower diameter) with the wall of the hole.

For most purposes each concrete dust sample can be associated to a depth from the concrete surface equivalent to the mid point of its drilling step, when plotting the concentration profile. Nevertheless, when it is necessary a more careful calculation of the depth of each sample, the geometry of the drilled hole must be taken into account [4]. The procedure for these calculations, which considers the geometry of the bit tip, is outlined in the Annex of this recommendation. This may be of interest for instance when the concentration profile is to be used as an input of a mathematical model for deriving transport parameters or predicting the corrosion initiation time.

5.1.1 Dry drilling equipment and procedure

The minimum equipment needed is:

- Drilling machine.
- Drill bits of appropriate diameter (for concrete minimum 18 mm).
- Dust collector.
- Compressed air or air blower.
- Slide gauge or similar.

In both field and laboratory cases the procedure will be the following:
1.a.– Field. Selection of the testing area. The locations shall be marked on a sketch of the structure. Paint or similar and any fouling must be removed from the surface.
1.b.– Laboratory. The area will be the top or bottom of the core or specimen. The selected area has to be that within a diameter approximately 10 mm less than the full diameter or size of the specimen (Figure 2)
2.- Placing and fixing of the machine and dust collector.
3.- For each depth interval, concrete dust is gathered in separated marked plastic bags.
4.- The drill hole, drill bit and dust collector shall be cleaned between each step interval by use of compressed air or air blower.
5.- For each sample of dust collected, the depth is measured at the center of the hole by a slide gauge or similar with an accuracy of at least 0.1 mm.
6.- The concrete dust collected in plastic bags is marked with structure denomination, location, depth interval and date.
7.- The total profile will consist of minimum 5-8 steps.

5.2 Grinding method

This procedure consists in collecting the dust obtained when grinding thin successive layers of concrete parallel to the exposed surface. The grinding is performed by using a turning lathe with an adequate working tool (on concrete specimens or cores taken from structures), or with especially constructed commercial devices equipped with a diamond bit rotating horizontally, that may be used on concrete specimens or cores and on real structures. This grinding method may be considered as a sampling procedure with high spatial resolution, since it allows obtaining powdered samples corresponding to thin layers of 0.5-2 mm thickness. The samples can be easily assigned to a depth from surface corresponding to the centre of the grinding step. The grinding areas are usually higher than 40 cm^2, in order to get large enough samples (minimum 5 g), thus avoiding heterogeneity problems.

5.2.1 Grinding equipment and procedures

Equipment

In the case of real structures two sampling strategies may be used: to take cores representative of the selected areas, which will be ground later at laboratory (with a lathe or with a grinding device); or to use the especial grinding device on-site. The adequate diameter of the cores or specimens is normally about 10 cm, although it depends on the grinding device used.

The equipment needed is:

- Grinding device or turning lathe.
- Appropriate grinding tools (bits).
- Dust collection system.
- Compressed air or air blower.
- Slide gauge or similar.
- General equipment for extracting cores from a concrete structure, and equipment for dry cutting concrete specimens, (in the case of using a lathe or when it is decided to grind at laboratory).

Procedures

In field sampling (grinding on-site), the steps will be:

1- Selection of testing areas, paint or similar and any fouling must be removed from the surface.
2- Fixing of the grinding equipment to the concrete structure surface. This ensures a perpendicular grinding to surface.
3- Preparation of plastic bags for collection of dust.
4- Grinding of samples in the predetermined layers or depth steps (it must be ensured that at least 5 g of dry concrete dust is collected from each layer).
5- Between the grinding of each layer, the grinding equipment and grinding area must be thoroughly cleaned by use of compressed air or air blower.
6- For each sample of concrete dust collected, the depth step below the surface is calculated as the average of four measurements with a slide gauge or similar of penetration depth with an accuracy of least 0.1 mm.
7- The concrete dust of each layer is collected in marked plastic bags to be analyzed for chloride concentration. The marks of each bag will consist on the structure denomination, location, depth interval and date.

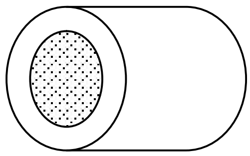

Figure 3. The grinding area must be slightly smaller than total face, in the case of a concrete core or specimen.

In laboratory sampling, (on specimens or cores taken from structures), the steps will be:

1- The area of the grinding will be the top or bottom face of the core or specimen. The grinding will be performed within a diameter approximately 10 mm less than the full diameter or size of the sample, see Fig. 3.
2- Preparation of the specimen or core for grinding. Usually the specimen must be cut to fit into the grinding device or lathe housing. It is preferably to perform all these cuttings in dry conditions, without using water as a lubricant, in order to prevent the washing out of the soluble chlorides.
3- Fixing of the specimen to the grinding device or lathe. This ensures a perpendicular grinding to surface.
4- Preparation of plastic bags for collection of dust.
5- Grinding of samples in the predetermined layers or depth steps (it must be ensured that at least 5 g of dry concrete dust is collected from each layer).
6- Between the grinding of each layer, the grinding equipment and grinding area must be thoroughly cleaned by use of compressed air or air blower.
7- For each sample of concrete dust collected, the depth step below the surface is calculated as the average of four measurements with a slide gauge or similar of penetration depth with an accuracy of least 0.1 mm.

8- The concrete dust of each layer is collected in marked plastic bags to be analyzed for chloride concentration. The marks of each bag will consist on the structure denomination, specimen denomination, depth interval and date.

The procedures for extracting cores from a concrete structure and for cutting concrete cores or specimens are not described since they are routinely performed by all control laboratories specialized in the study of structures. An important precaution when extracting the cores is to protect them immediately from drying, and to proceed as soon as possible to the grinding process, in order to avoid chloride spatial redistributions.

5.3 Cutting and crushing method

This procedure can be applied only at laboratory, since it starts with a concrete cast specimen or with a core taken from a real structure. The specimen is dry cut in slices of adequate thickness, which will be later crushed into powder to get the samples to be analyzed for their chloride contents, see Fig. 4. The number and thickness of the slices must be decided by estimating the penetration depth reached by the chlorides into the structure, and by considering the desired spatial resolution. The normal thickness of the slices is about 5-10 mm, although it is possible to cut thinner slices of about 2 mm. Taking this into account the spatial resolution of this method is comparable to that of the dry drilling method. On the other hand the samples can be more easily assigned to a depth corresponding to the centre of the slice. Another inconvenience of this method is the loss of material due to the cutting process. The habitual diamond saws used for dry cutting concrete specimens have a thickness of about 5 mm. This implies that 5 mm of concrete depth are lost for each slice cut. The size of the concrete powdered samples must be large enough to guarantee a representative profile and to enable an accurate determination of the chloride content by the corresponding chemical procedure (minimum 5 g of dust for each sample). The crushing of the slices is performed manually with a mortar and pestle or mechanically with ball or ring mills.

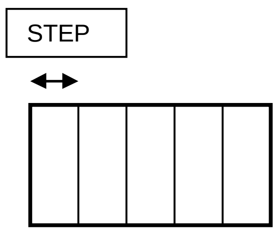

Figure 4. Slices to be cut from a specimen and crushed later for gaining the concrete dust samples.

The risk of modifying the real chloride content of the samples comes from the fact that the coring procedure at structures is performed with water lubrication, which may wash out the soluble chlorides. For this reason it is recommended to eliminate mechanically by dry cutting or filing away the peripheral portions of the extracted cores, which are the zones more susceptible to be washed out, before starting the cutting process.

5.3.1 Cutting and crushing equipment and procedures

Equipment

In the case of real structures it is mandatory to take cores representative of the selected areas, which will be cut and crushed later at laboratory. There is no limitation for the diameter of the cores or specimens, but too small diameters and too thin slices will produce samples of lower size than required. Habitual core diameters are about 5-10 cm.

The equipment needed is:

- General equipment for extracting cores from a concrete structure.
- Diamond saw to dry cut the concrete slices.
- Mechanical equipment for filing away the washed peripheral zones of the cores.
- Crushing equipment, mortal and pestle, ball mill, ring mill, etc.
- Slide gauge or similar.

Procedure

1- Preparation of the specimen or core. If necessary eliminate mechanically by dry cutting or filing away the peripheral portions of the extracted cores.
2- Mark the desired points of cutting at the lateral face of the core or specimen, taking into account the material eliminated by the saw at each cutting.
3- Fixing of the specimen or core to the cutting machine. This ensures a perpendicular cutting to the specimen axis.
4- Preparation of plastic bags or containers for the slices.
5- Perform the dry cutting of each slice. Each slice must be marked to allow its identification.
6- Measure the thickness of each slice, performing four measurements with a slide gauge or similar, with an accuracy of 0.1 mm. Calculate the depth step corresponding to each slice, taking into account the depth lost in each cutting. Store the slices in their bags or containers, marking them with the structure denomination, location, depth interval and date.
7- Preparation of plastic bags for collection of dust.
8- Crushing of the slices. Normally this operation is performed in at least two steps: a manual rough crushing with a mortar and pestle; and a posterior milling to the desired grain size using a ball or ring mill. The habitual chloride chemical analysis methods require the sample to be milled to pass through 0.16 or 0.32 mm sieves. The nature and composition of the milling elements and containers must be adequate for the hardness of the concrete under study.
9- Between the crushing and milling of each slice, all the equipment must be thoroughly cleaned.
10- The concrete dust of each slice is collected in marked plastic bags to be analyzed for chloride concentration. The marks of each bag will consist on the structure denomination, specimen denomination, depth interval and date.

An important precaution when extracting the cores from a structure is to protect them immediately from drying, and to proceed as soon as possible to the cutting process, in order to avoid chloride spatial redistributions.

6 ANNEX. BIT SHAPE GEOMETRIC CONSIDERATIONS FOR THE ASSIGNMENT OF DEPTH TO THE DRY DRILLED DUST SAMPLES

The accurate assignment of depth to each sample obtained by dry drilling needs a consideration of the geometry of the drilled hole, which depends on the geometry of the bit tip. While there are bits with almost flat tips, the more common masonry bits end with a conical tip, see Fig. 5. For the bits with flat tips each dust sample can be assigned to the centre of the depth step. Nevertheless, for a conically shaped bit tip the amount of dust removed is not the same all along the drilling depth step; and furthermore for the second and subsequent samples, concrete dust from depths lower than the nominal drilling step is obtained, see Figs. 6A and 6B. This leads to the conclusion that each Cl^- concentration in the profile should be assigned to the gravity centre (GC) of the solid sample really taken in the drilling step, in order to avoid systematic errors by excess in the derivation of the ionic diffusion coefficients [4]. Figs. 6A and 6B show the positions of the GC of the dust samples obtained by drilling with a conically shaped bit of 18 mm diameter.

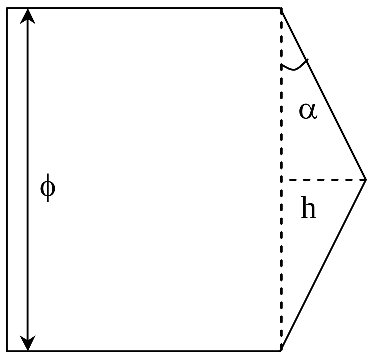

Figure 5. Ideal projection of a bore hole performed with a bit of diameter ϕ.

A method for calculating the GC of the samples obtained with a conically shaped drill bit was suggested [4]. The input data for these calculations are the thickness of the drilling steps (L) and the height (h) of the cone that constitutes the bit tip, see Fig. 5. The explicit formulae for the calculations of the positions of the GC of the first sample and the second and subsequent, (nth samples), which were not given before, are as follows:

First sample :
$$x_{GC} = \frac{\left(\frac{L^2}{2} + \frac{h^2}{4} - \frac{2Lh}{3}\right)}{\left(L - \frac{2h}{3}\right)} \qquad \textit{for } L \geq h \qquad (1)$$

Second and subsequent samples (nth sample)
$$x_{GC} = (n-1)L + \frac{L}{2} - \frac{2h}{3} \qquad (2)$$

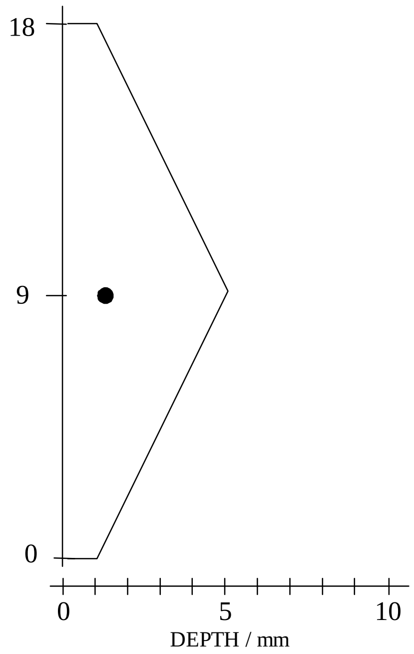

Figure 6A. Ideal geometric projection of the first 5 mm step sample, taken by drilling with a 18 mm diameter bit from 0 to 5 mm depth from surface. The solid point indicates the approximate position of the gravity centre.

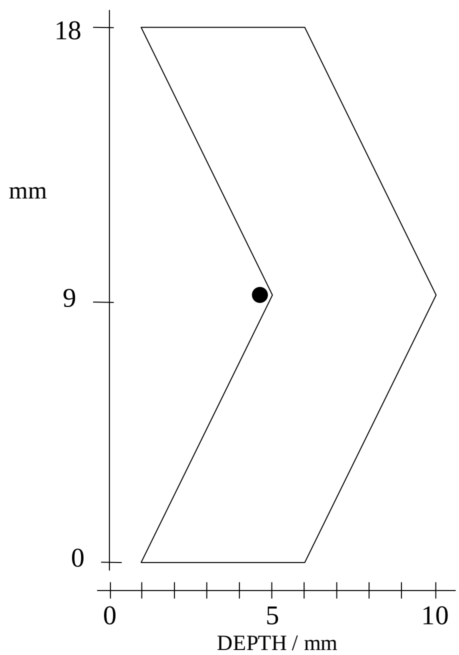

Figure 6B. Ideal geometric projection of the second 5 mm step sample, taken by drilling with a 18 mm diameter bit from 5 to 10 mm depth from surface. The solid point indicates the approximate position of the gravity centre.

Where x_{GC} is the depth from surface of the concrete dust sample, which coincides with the gravity centre of the sample taken by dry drilling. It is important to bear in mind that Equation (1) is only valid if the thickness of the drilling step, L, is higher or equal to the bit cone height, h. Equation (2) is valid if L is constant for all the drilling steps.

Table 2 contains the positions of the GC of the concrete dust samples obtained by dry drilling with conically shaped bits of usual diameters, from 12 to 22 mm. These positions have been calculated with Equations (1) and (2).

Table 2. Positions of the gravity centres, (depth from surface), of samples obtained by dry drilling.

Bit diameter, ϕ (mm)	Bit tip cone height, h (mm)	Drilling depth step, L (mm)	Gravity centre of solid sample (mm)		
			1st step	2nd step	3rd step
12	3	5	1.6	5.5	10.5
		10	4.0	13.0	23.0
18	4	5	1.4	4.8	9.8
		10	3.7	12.3	22.3
		20	8.7	27.3	47.3
20	4.5	5	1.3	4.5	9.5
		10	3.6	12.0	22.0
		20	8.5	27.0	47.0
22	5	5	1.3	4.2	9.2
		10	3.4	11.7	21.7
		20	8.4	26.7	46.7

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