### **Admixtures and Special Concretes**

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## Lecture -17

# **Chemical Admixtures: Standards**

# **ASTM C494:**

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A superscript epsilon (ɛ) indicates an editorial change since the last revision or reapproval ε1 NOTE-Table 1 was editorially corrected in August 2022.

This standard has been approved for use by agencies of the U.S. Department of Defense.

#### 1. Scope\*

1.1 This specification covers materials for use as chemical admixtures to be added to hydraulic-cement concrete mixtures for the purpose or purposes indicated for the eight types as follows

- 1.1.1 Type A-Water-reducing admixtures,
- 1.1.2 Type B-Retarding admixtures, 1.1.3 Type C-Accelerating admixtures,
- 1.1.4 Type D-Water-reducing and retarding admixtures,
- 1.1.5 Type E-Water-reducing and accelerating admixtures,

1.1.6 Type F-Water-reducing, high range admixtures, 1.1.7 Type G-Water-reducing, high range, and retarding

- admixtures, and
- 1.1.8 Type S-Specific performance admixtures.

1.2 Unless specified otherwise by the purchaser, test specimens for qualifying an admixture shall be made using concret- comparisons shall be made. ing materials as described in 11.1 - 11.3.

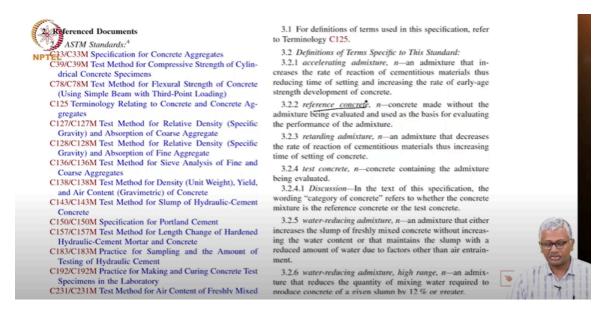
Note 1—As discussed in Appendix X2, it is recommended that, whenever practicable, supplementary tests be made by the purchaser using the cenent, pozzolan, aggregates, air-entraining admixture, and the mixture proportions, batching sequence, and other physical conditions proposed for the specific work because the specific effects produced by

Table 1 demonstrates that the admixture meets the requirements of this specification. Proof of compliance shall be based on comparisons of the average test results from the batches of test concrete and the average test results from the batches of reference concrete. Admixtures (except for Types B, C, E, and S) shall qualify for provisional compliance if the time of setting, length change, and durability factor meet the physical requirements and any of the alternative compressive strength requirements shown in parentheses in Table 1 are met through the date of provisional acceptance (see Note 4). If subsequent test results at six months or one year fail to meet the requirement of at least 100 % of reference strength, the provisional compliance of the admixture to this standard is withdrawn and all users of the admixture shall be notified immediately. Uniformity and equivalence tests of Section 6 shall be carried out to provide results against which later

Note 4-Allowing for provisional compliance while retaining los NOTE 4—Allowing for provisional compliance while retaining longer term compressive strength requirements promotes more rapid qualification of new materials, but also provides assurance that new admixture technologies will not exhibit unexpected longer term performance. The alternative compressive strength requirements in Table 1 are based on statistical analysis of 103 Specification C4040/404M evaluation tests. The

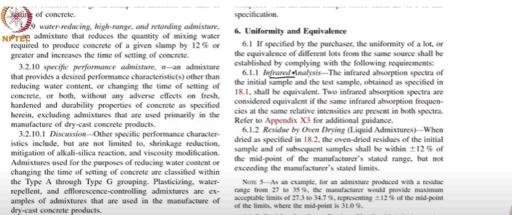
Okay, so as I was saying in the past there were 7 types of chemical admixtures that were specified in ASTM C494. Type A is water reducing, type B is retarding, type C is accelerating, type D is water reducing and retarding that means it has got a double functionality and that is quite easy to imagine because most of the carboxylic acids and lignosulphonates act as both water reducers and retarders. Type E is water reducing and accelerating, so obviously this is not from the same molecule but you need to have 2 molecules which can actually work together to give that kind of performance. Type F is water reducing high range super plasticizer and type G is a retarding super plasticizer, water reducing high range and retarding. Now what we have also brought on additionally is this type S or special performance admixtures. Now it is a very general name that has been given here, it does not really qualify any specific characteristics that need to be met by the admixture. I will show you the table later which essentially captures the kind of performance you can get from all of these admixtures.

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So I will request you to go through the details on your own but what I wanted to show you is when you prepare a reference concrete like here they have reference concrete, concrete made without the admixture being evaluated and used as the basis for evaluating the performance of the admixture. So when you make a reference concrete and you make a concrete with the admixture the differences or the alterations in performance should be as per the minimum standards that are prescribed in this specification. So let us talk about that briefly.

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6.1.3 Residue by Oven Drying (Nonliquid Admixtures)— When dried as specified in 18.3, the oven-dried residues of the initial sample and of the subsequent samples shall be within a range of variation not greater than  $\pm 4$  percentage points.

6.1.4 Relative Density (Specific Gravity) (Liquid Admixtures)—When tested as specified in 18.4, the relative



Ordering Information

 The purchaser shall specify the type of chemical admixture desired, and in the case of a Type S admixture the specific

performance characteristic(s) required.

So again one more important thing are the aspects to check in an admixture for its uniformity and equivalence. You need to do an infrared analysis which we call as Fourier transform infrared spectroscopy. The idea here is to actually obtain the actual organic signature of this compound. The infrared analysis helps you to understand whether the organic compounds are as they have been prescribed in the formulation. Of course nobody would, no construction chemical manufacturer will obviously reveal their formulation entirely but there will be some signature elements that will be present in most of these compounds that can be detected with the help of IR analysis.

Residue by oven drying is a very important test to be conducted because we know that most of these liquid admixtures are solutions of 30 or 40% of the solid component in water. So that is the other aspect. In some cases there are non-liquid admixtures which may also have some moisture which is actually absorbed or absorbed in the formulation. So here again such admixtures also you may actually want to do the residue by oven drying. Specific gravity or relative density is also measured typically using a hydrometer just like what you do for soils. You must have used the hydrometer for sedimentation analysis, same thing. You can use the hydrometer for detecting the specific gravity.

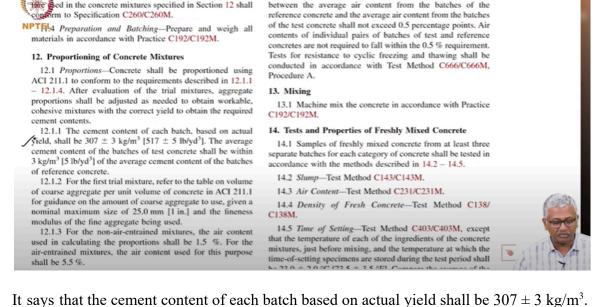
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TEL	Type A, Water- Reducing	Type B, Retarding	TABLE 1 Physic Type C, Accelerating	Type D, Water- Reducing and Retarding	Type E, Water- Reducing and Accelerating	Type F, Water- Reducing, High-Range	Type G, Water- Reducing, High-Range and Retarding	Type S Specific Perfor- mance	
Water content, max, % of reference <sup>4</sup> Time of setting, allowable deviation from reference, h:min:	95			95	95	88	88		
Initial: at least not more than	1:00 earlier nor 1:30 later	1:00 later 3:30 later	1:00 earlier 3:30 earlier	1:00 later 3:30 later	1:00 earlier 3:30 earlier	1:00 earlier nor 1:30 later	1:00 later 3:30 later	1:00 earlier nor 1:30 later	
Final: at least not more than	1:00 earlier nor 1:30 later	3:30 later	1:00 earlier	3:30 later	1:00 earlier	1:00 earlier nor 1:30 later	3:30 later	1:00 earlier nor 1:30 later	
Compressive strength, min, % of reference: <sup>C</sup>									
1 day						140	125		
3 days	110	90	125	110	125	125	125	90	and a
7 days	110	90	100	110	110	115	115	90	
28 days	110 (120) <sup>D</sup>	90	100	110 (120) <sup>D</sup>	110	110 (120) <sup>D</sup> 1	110 (120) <sup>D</sup>	90	· Des
90 days	(117) <sup>D</sup>	n/a	n/a	(117) <sup>D</sup>	n/a	(117) <sup>D1</sup>	(117) <sup>D</sup>	n/a	(256)
6 months	100 (113) <sup>D</sup>	90	90	100 (113) <sup>D</sup>	100	100 (113) <sup>01</sup>	100 (113) <sup>D</sup>	90	
1 year	100	90	90	100	100	100	100	90	

So here this is the main table that we need to look at. Table 1, physical requirements. So you have designed a reference mix in ASTM very specifically. So it gives you a clear idea about what the reference mix should be. Just one minute, let me get to that section here. It gives you the grading of the aggregate to be followed for the reference mix and then it also gives you proportioning, mixture proportioning of the concrete mixtures.

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3 Air-Entraining Admixture-The air-entraining admix-

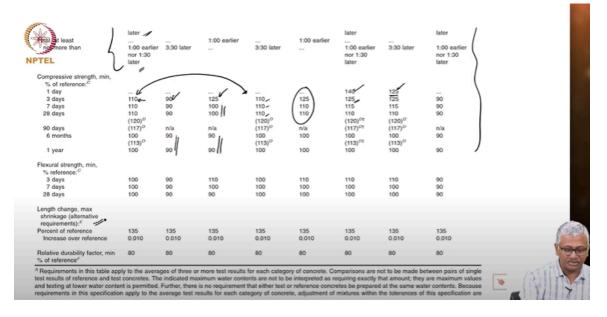


concrete and test concrete shall be 6.0 ± 1.0 %. The difference

The average cement content of each batch based on detail yield shall be  $50^{\circ} \pm 5^{\circ}$  kg/m<sup>3</sup>. The average cement content shall be within 3 kg/m<sup>3</sup> of the average content of the batches of reference concrete. So you prepare a reference concrete with this kind of a formulation with cement content of so much. You can then include for instance the air entraining agents. If you want to have an air entrainment in your concrete and you adjust the water content to obtain a slump of 90 ± 15 mm. Around 100 mm slump is what you design the reference concrete for. So based on that water content you need to then do an estimation of the water reduction that happens when you use the water reducing admixtures. So just coming back to this table again. It clearly says that for type A water reducing admixture the water content maximum should be 95% of the reference mix. That means you should get at least 5% water reduction. Similarly type D, type E both are also regular water reducers so 5% water reduction is what is desired from these chemicals. Then you have type F where the minimum is 12% water reduction, 12% or more and type G also is 12% or more. Type S there is no specific performance with respect to water reduction. It may have other functionalities shrinkage. All of those other things could be there.

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PTEL	Type A, Water- Reducing	Type B, Retarding	Type C, Accelerating	Type D, Water- Reducing and Retarding	Type E, Water- Reducing and Accelerating	Type F, Water- Reducing, High-Range	Type G, Water- Reducing, High-Range and Retarding	Type S Specific Perfor- mance	
Water content, max, % of reference <sup>4</sup> Time of setting, allowable deviation from reference.	95			95	95	88	88	-	
h:min:									
Initial: at least not more than	1:00 earlier nor 1:30 later	1:00 later 3:30 later	1:00 earlier 3:30 earlier	1:00 later 3:30 later	1:00 earlier 3:30 earlier	1:00 earlier nor 1:30 later	1:00 later 3:30 later	1:00 earlier nor 1:30 later	
Final: at least			1:00 earlier		1:00 earlier	_			
not more than	1:00 earlier nor 1:30 later	3:30 later	-	3:30 later	_	1:00 earlier nor 1:30 later	3:30 later	1:00 earlier nor 1:30 later	
Compressive strength, min, % of reference: <sup>C</sup>									
1 day		6	100			140	125		
3 days	110	90	125	110	125	125	125	90	
7 days	110	90	100	110	110	115	115	90 90	
28 days	(120) <sup>D</sup>	90	100	110 (120) <sup>D</sup>	110	(120) <sup>01</sup>	(120) <sup>D</sup>	90	
90 days	(120)	n/a	n/a	(117)0	n/a	(120) (117) (117) (117)	(120) <sup>0</sup>	n/a	
6 months	100	90	90	100	100	100	100	90	
o monuta	(113)0			(113)0	100	(113)01	(113)0	50	
1 year	100	90	90	100	100	100	100	90	
. your									
Flexural strength, min,									
% reference:C									_
3 days	100	90	110	100	110	110	110	90	1
7 days	100	90	100	100	100	100	100	90	
28 days	100	90	90	100	100	100	100	90	



Now when you add the admixture type A water reducing admixture you have to ensure that the admixture is formulated such that there is not much change in the initial or final setting time. It says not more than 1 hour earlier nor 1.5 hours later the initial setting time as well as final setting time. Why is that? Because we do not want from type A admixture we do not want any retarding or accelerating effects.

Now if you look at type D water reducing and retarding it says that the initial should be at least 1 hour later but not more than 3.5 hours later. So if the reference concrete sets at 4 hours your concrete with the retarding admixture should at least take 5 hours to set but not more than 7.5 hours. So that is how you will actually read this entire standard.

Then coming to the hardened concrete properties compressive strength minimum at one day only for the high range water reducers there is a requirement but for all the other admixtures the requirement is at later ages. For instance water reducing admixture should at least lead to a 10% enhancement of the strength at 3, 7 and 28 days. Of course they go all the way up to 1 year if you really have to get your admixture certified the certifying laboratory has to actually conduct the tests for a period of 1 year. So this is a long term investigation. So you cannot just go to a lab and say I need a certificate of ASTM type A it will take at least 1 year to get that.

Now type B retarding we know that retarding admixture will lead to a reduction in the initial rate of strength gain because of that you are permitted 90% of the strength of reference mix at 3, 7 and 28 days. So you really do not want a very high level of performance with respect to strength from the plain retarding admixture. Mind you plain retarding means it does not have any water reduction capability like if you use a zinc compound or boron compounds which are not the organic chemicals which are also functioning as water reduces.

Type C accelerating you have to obviously get a benefit in the early strength. So at 3 days you need 25% higher strength in the reference but later you are okay with lesser or equal strength and in the long term even lesser strength as compared to the reference mix. At 6 months or 1 year you are okay with 90% of the reference because as I mentioned earlier when you accelerate you are likely to get a loss in the long term strength. So up to 10% strength loss is allowable when you have an accelerating admixture.

Water reducing and retarding again the numbers are quite similar to what you have for a typical normal water reducer. After reducing and accelerating you have the accelerating component at 3 days but after that it is mostly equivalent to a normal water reducer because the accelerator will work primarily for the early strength later age strengths are determined by the water reduction.

Now again with respect to type F water reducing high range you have to have 40% more strength at 1 day, 25% more strength at 3 days, 15% at 7 days and after that it matches more or less what you have with your water reducing admixture. All this has to be achieved with 12% minimum water reduction. So you can also design your concrete mix with 80% of water content of the reference that means 20% water reduction. So all that will be determined by what is the recommended dosage of the admixture to be used to provide that level of water reduction and that is provided by the manufacturer. So when the laboratory certifies these products as type A or type F they are certifying it based on the range of dosages recommended by the manufacturer. So when you try to use in a real project dosages that are not in the recommended range there is no guarantee of the performance. So the admixture company will guarantee the performance only in that range. So it is very important to pay attention to that.

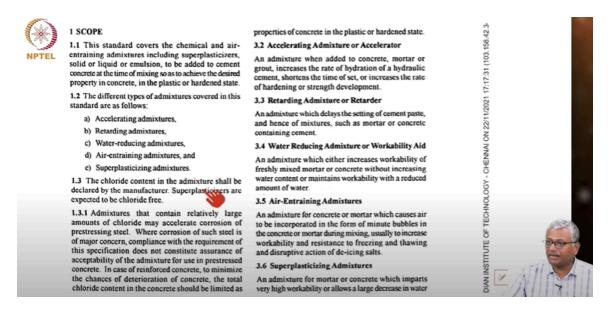
Type G water reducing high range and retarding your requirement for 1 day strength is not as stringent as type F but you still need 25% higher strength. That is obviously the water reducer which is reducing water to a large extent at least 12% leading to that higher strength but after that more or less it matches the type F requirement. Now when you have a specific performance to be given to your concrete it may have a range of characteristics that you are looking at but at least it has to satisfy all of these characteristics with respect to strength and setting time. There are no demands made out of the concrete with specific performance. For instance viscosity modifying admixture if you add to your concrete you need some rheology modification of your concrete. So that kind of concrete should also satisfy the setting time and strength characteristics. Mind you this is the setting time of the concrete not the setting time of the cement. There is a difference.

Again similar requirements are given for flexural strength. Requirements are also given for length change, maximum shrinkage requirement is given. Percentage of increase over reference is 35% in most of the cases that means you are allowed a slightly higher level of shrinkage when you use these chemical admixtures. Increase over reference in absolute terms is given here.

Durability factor is a test that you do for resistance to freezing and thawing. This is something that we will discuss in our chapter on air-entering admixtures. The durability factor is the test that you do for freezing and thawing. There your concrete with chemical admixtures needs to have at least 80% of the durability factor of the reference. We will talk about this durability factor later on. So these are the requirements given. When the concrete is designed with this kind of a proportion, whatever is given here with  $307\pm3$ kg/m<sup>3</sup> and a water content that is sufficient to get you 100 mm slump. This is a standard test done for the specification. For your job site requirement you may have obviously other mix designs. Cement content water binder ratio will be depending on the strength and other performances that you desire for your concrete. You can then read the remaining standard in much more detail and try to understand what the differences are. Test methods are also given for infrared analysis, for liquid admixtures, for determination of the solids content and so on. All of these test methods are already given here in the standard so you can refer to them at your own leisure.

# IS 9103:

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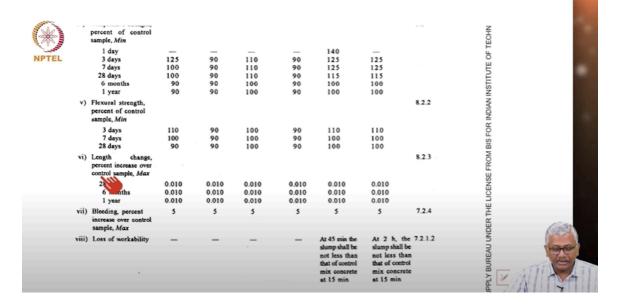
So I will move on to the other code that is the IS 9103. So this one IS 9103 again it is a 1999 code and it was reaffirmed in 2013 and then in 2018. Reaffirmed means without any changes we have adopted the same standard again. Whenever a change is made there is an amendment that is added to the standard. So IS 456, 2000 if you look at there are some 7or 8 amendments that are already there and these amendments are there in the beginning of the standard. So in this specification it is more or less similar to what you saw with ASTM. Again they define the different types of admixtures as accelerating, retarding, water reducing, air entraining and super plasticizing. So again one important aspect is that super plasticizers are expected to be chloride free. They have specified here they also say in ASTM I have not indicated that segment but it is very important for us to understand that admixtures should not be the source of bringing in the chlorides that cause corrosion.

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			Table 1A	Physical Re	quirements				31/03
		(Clause 4)							PT 00
SI No.			Retarding Admixture		Air-Entrain- ing Admixture	Admixtur	lasticizing e (for Water- Concrete Mix)	Test Ref	CHENNAI ON 22/11/2021 17:17:31 (103.158.42.34) VALID UPTO3102/
						Normal	Retarding Type		34
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	8.42
i)	Water content, percent of control sample, Max	lu	-	95	-	80	80	7.2.5	1 (103.15
ii)	Slump	-	-	-	-	belo	w that of the mix concrete	7.2.1	1 17:17:3
iii)	Time of setting, allowable deviation from control sample							7.2.3	2/11/202
	hours:								Z
	Initial								2
	Max	- 3	+ 3	# 1	_		+4		2
	Min	- 1	+1		_	+ 1.5	+1		<u><u> </u></u>
	Final								0
	Max	- 2	+ 3	#1		± 1.5	13		6
	Min	- 1	+1	-	_		_		DI TIT
iv)	Compressive strength, percent of control							8.2.1	LECHNOLOGY

So again requirements for admixtures are given in a table similar to what you have in ASTM. So as in ASTM 95% is the percentage of water with respect to the control or reference sample for a water reducing admixture. For super plasticizing admixture they have directly gone to 80% that means they want 20% water reduction in IS 9103. So they have adopted a slightly higher level of water reduction to be desired. Again similar qualifications, time of setting, allowable deviation from control sample, maximum or minimum deviation from the initial and final setting time are also provided similar to what you have in ASTM. Strength requirements are more or less similar to what you have with ASTM, not much different.

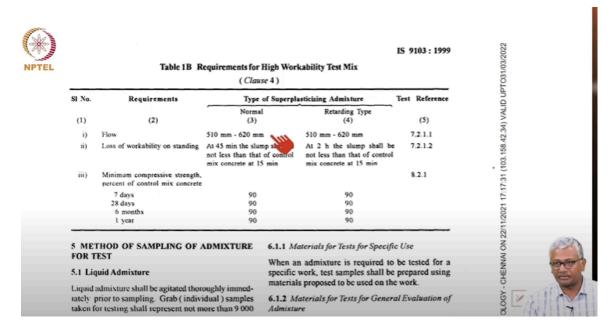
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And again length change, percentage increase over control sample is given here and bleeding percentage increase of control sample this is an extra requirement, bleeding. So what happens is when you start using admixtures some of the water may get freed up and start bleeding out. So you need to control that bleeding to ensure that you have a controlled bleeding and you do not have excess bleeding when these admixtures are used. There is a limit to how much percentage increase you can actually have for the bleed water as compared to the reference sample. But what you will see is in most cases because we are already reducing the water by using these chemicals the bleed water is automatically also reduced as compared to the reference sample.

In a couple of cases for the super plasticizing admixture which is normal or retarding type corresponding to type F or type G of ASTM. Here they also specify a test for loss of workability. Now of course the design of concrete for assessing loss of workability is not the same as design of concrete for assessing water reduction. So you need to design it differently. They have specific ideas as to how we need to do the design. So it says that

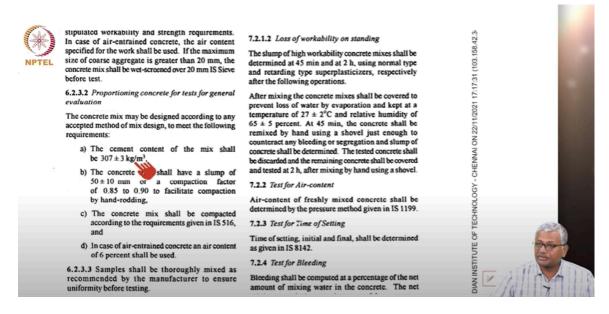
45 minutes the slump shall not be less than that of control mix concrete at 15 minutes. That means whatever reference concrete slump is at 15 minutes your super plasticized concrete should be the same at 45 minutes. Here with retarding type at 2 hours the slump should be equal to that of the control concrete at 15 minutes.



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So here requirements for a high workability test mix. So here they are also identifying the fact that these chemicals are used for producing self-compacting concrete. So flow needs to be measured in self-compacting concrete or slump flow. For normal and retarding types the flow requirement is 510 to 620 mm. Loss of workability again is similar to what I already described in the previous table for normal workability and minimum compressive strength here requirement is only 90% of your reference concrete because when you produce a high workability mix you do not reduce the water you maintain the water binder ratio but you add the plasticizer to start making the concrete flowable that is the idea.

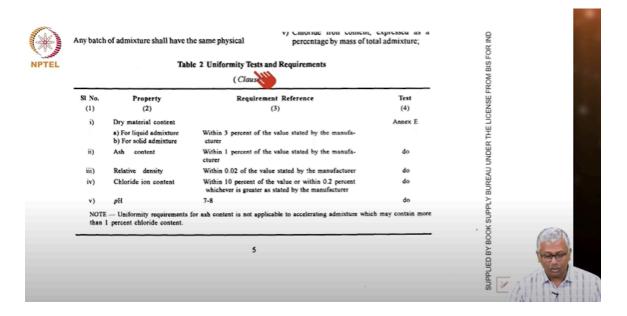
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Preparation of concrete as I said the mixture is almost similar cement content is again  $307\pm3$  kg/m<sup>3</sup>. Here the concrete needs to be designed for a slump of  $50\pm10$  kg/m<sup>3</sup> that is again different from that in ASTM. Here we are looking at 50 in ASTM it is 100 so there is a difference there.

So again they also say that in case of air entrained concrete then air content of 6% shall be used, which is the same in ASTM also. So that is the kind of reference mix that they want to make and based on the water reduction you are basically getting the different performance requirements.

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And some of the tests are described in the same standard, some of them are marked to other standards so you can see. They also talk about uniformity tests again. I did not show you this clause in ASTM. It is also there because on a job site the admixture will be supplied on a regular basis. You will not supply everything at once and it will not be the same lot. So when you get different lots of the admixture you need to test them to make sure that the chemical is uniform and to do the test you need to actually do all of these dry material content, ash content, relative density, chloride content and pH. So what they say is the pH of course all the other materials dry material content within 3% of the value stated by the manufacturer. So the manufacturer's data sheet has a certain value when you do the testing it should be within 3% of that. Similarly ash content should be within 1% of the value suggested by the manufacturer. So you need to look at testing your samples of admixture that have been received for production. Just like you test cement, what test you do on cement typically when you get new cement on the job site.

For any cement what minimum test you need to do? Fineness consistency, initial setting time, final setting time and compressive strength. Soundness also but most site labs may or may not be doing soundness but at least they will do setting time and compressive strength.

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()	ANNI ( Note under	T031/03/	
NPTEL	TEST FOR RESISTANCE OF CONCRETE	an ar	
	B	n1 = fundamental transverse frequency at zero cycle of freezing and thawing; and	2.34) VAU
	B-1 The freezing and thawing cycle shall consist of alternately lowering the temperature of the specimens	n2 = fundamental transverse frequency after C cycle of freezing and thawing.	3,158,42
	from $4 \pm 1^{\circ}C$ to $-18 \pm 2^{\circ}C$ and raising it from $-18 \pm 2^{\circ}C$ to $4 \pm 1^{\circ}C$ in not less than 2 h nor more than 5 h. The freezing and thawing can take place either in (i) water, or (ii) freezing in air and thawing in water.	The relative durability factor shall be calculated as follows: $DF( \text{ or } DF_1) = PN/300$	ON 22/11/2021 17:17:31 (103.156.42.34) VALID UPT 031/03/
	B-2 The test for fundamental transverse frequency shall be conducted on concrete prism specimens (100 mm × 100 mm × 400 mm or 150 mm × 150 mm ×	$RDF = DF/DF_1 \times 100 \label{eq:RDF}$ where	2/11/2021 1
	600 mm ) or on concrete cylinder specimens (150 mm in dia × 300 mm length ). The test shall commence at	DF = durability factor of the concrete containing the admixture under test,	ON 22
	the age of 14 days ( water-curing till then ) by electro- dynamic method in accordance with IS 516.	DF <sub>1</sub> = durability factor of the concrete containing the reference admixture,	ENNA
	The relative dynamic modulus of elasticity shall be calculated as follows: $P_6 = n_2^{-2}/n_1^{-2} \times 100$	P = relative dynamic modulus of elasticity in percentage of the dynamic modulus of elasticity at zero cycle (values of P witt be 60 or greater), and	ECHNOLOGY - Ch
	where	N = number of cycles at which P reaches 60 percent or 300 if P does not reach	LECH

So again you can go through this standard at your leisure to try and understand. So again they have also talked about tests of resistance of concrete against freezing and thawing. Interestingly, it turns out that the chambers that are required for this experiment are perhaps not even available in India. I am not very sure I have not seen any lab which has this chamber available but I may be wrong. There may be one or two private labs that have this chamber. So the freezing and thawing test is not really performed much in India because except for certain regions we really do not have a problem of the kind of freezing and thawing climates that really cause problems of the concrete and that I will show you when we talk about air and training agents.

So again it gives you other methods also determination of flow of concrete of high workability essentially it is describing the slump flow test and the flow drop test also. So all of these are test to be used for high flowable concrete and test for bleeding of concrete. So I will close this and then move on to the next chapter. So the idea is to pay attention to the details that are given there because very often on sites the kind of quality test that we do may or may not be adequate for the purposes of actually assessing the properties of the admixture. Addition to that you may also want to do the marsh cone test which we discussed earlier when it is a super plasticizer it is always better to do the marsh cone test because that helps you assess very quickly what is the compatibility of this admixture with the cement that you are using for the project or if the cement source changes again marsh cone is very important to be done.