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MOONEY VISCOMETER

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Future Foundation

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MOONEY VISCOMETER

SPECIFICATIONS

1.	**Mooney measuring range**	:	0 to 200 MU (4 digits)
2.	**Pre-heat time**	:	0 to 10 minutes
3.	**Mooney measurements**	:	Choice of 3 readings
4.	**Scorch points**	:	Choice of 4 readings
5.	**Rotor size**	:	Large and small
6.	**Rotor speed**	:	2.0 ± 0.02 RPM
7.	**Drive Motor**	:	Synchronous geared motor
8.	**Temperature**	:	PT100 Platinum resistors Microprocessor controlled. Calibrated range 50 to 200°C. Independent Upper and Lower Platen Control.
9.	**Temperature Control**	:	Accuracy ± 0.5°C.
10.	**Torque transducer**	:	Reaction torque sensor. Four Arm temperature compensated semi-conductor strain gage bridge.
11.	**Recording & Display**	:	Computer controlled testing. Directly on-line display on VGA Monitor, Memory storage of data. Automatic computation of results. Multiple display of graphs. Statistical Analysis. Statistical Quality Control.
12.	**Printed Data**	:	80 column
13.	**Electric Power Supply**	:	AC 175 - 275V, 50 Hz, 20 Amp. Max.
14.	**Compressed air supply**	:	60 psi (4.2 kg / sq.cm) minimum operating pressure controlled by external regulator with gauge.
15.	**Environment**	:	Dust free reasonably controlled ambient temperature and humidity.
16.	**Dimensions**	:	A. Main Panel -63 x 26 x 23 inches B. Computer System - Table Top.

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Future Foundation

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MOONEY VISCOMETER

INTRODUCTION

MOONEY VISCOMETER: It is an instrument consisting of a motor driven disk within a die cavity formed by two dies maintained at specified conditions of temperature and die closure force. It measures the effect of temperature and time on viscosity of rubber or compound.

MOONEY VISCOSITY: It is a measure of the viscosity of a rubber or compound determined in a Mooney shearing disk viscometer. It is indicated by the torque required to rotate the disk embedded in a rubber/compound specimen and enclosed in the die cavity under specified conditions.

Viscosity of rubber or compound plays vital role in deciding its processing behaviour. In Rubber Industry rubber or compound have to undergo various processing before it can be vulcanized into its final form.

Deviation in viscosity of the compound will critically alter its processibility specially in terms of calendering, extruding or injection moulding. It is necessary that viscosity parameter be maintained within specified limits. To do this Mooney Viscometer is of vital importance. In fact viscometer has manifold advantages.

APPLICATIONS

1. PROCESSING BEHAVIOUR : The invention of new polymers and rubber chemicals compounder is faced within problem of choosing the right ingredient and its dose, specially with respect to its processing behaviour during calendering, extruding or injection moulding. As the processing behaviour of the compound changes with respect to its temperature, one has to predict how it is going to behave during processing. With the help of Mooney Viscometer one can predict its behaviour with respect to temperature and time during processing.

2. RESEARCH & DEVELOPMENT : The most tedious part in compounding is to develop a new compound to meet the desired results. It involves:

a) Defining required quality targets

b) Designing preliminary compounds, selecting specific ingredients and determination of dosage of each ingredient.

c) Checking the cost factor.

d) Testing each compound.

e) Re-designing the formulation till quality target is achieved. The process involves enormous work which is time consuming, expensive and requires skill. With the help of Mooney Viscometer, one cando all this exercise quickly with minimum wastage of materials.

QUALITY CONTROL: In order to produce consistent quality of Rubber products, it is of vital importance that compounded rubber is of consistent quality. As the compound is mixed in batches, batch to batch variation, if any, needs attention in controlling quality of each batch. If randomly selected batches are subjected to Viscosity analysis, one could in a large sample size, workout upper and lower control limits, range, mean and standard dedviation, with reference to Viscosity parameters. Each batch on testing can be classified on Pass/Fail criteria depending upon the quality control limits. Based on this, the internationally acceptable control called “Statistical Quality Control” (SQC) can be designed. Computerized data analysis system of Mooney Viscometer viz. “Viscosoft” is ideal in this regard. By this method, one can monitor easily consistency of each mix everyday.

CHOOSING THE CORRECT POLYMER: While developing new compound one has to choose the right polymer for the end use. Each polymer again is sub divided into various types depending upon their physical and chemical properties. Viscosity is of paramount importance before selecting its acceptability in the formulation. A high viscosity polymer can be selected where high filler loading is desired and a lower viscosity polymer is selected if other properties are important.

GRADING OF RUBBERS: Mooney Viscosity of the rubber is one of the important parameter in grading the quality of rubber. And it is also important parameter in choosing the rubber quality for application.

ECONOMICS: Everything said, but one may still be hesitant in investing in such instruments mainly because of its cost. Experience has shown that if Mooney Viscometer’s full potential is exploited with regards to few of various advantages mentioned above, the return is quite handsome. Improved quality, minimised wastage, optimised dosage of each ingredient, choice of right ingredients, controlled process certainly makes this instrument singularly ideal for any Rubber Industry.

MOONEY VISCOMETER

PRINCIPLE :

The viscosity of rubbers can vary dramatically with deformation rate, and thus their rheological properties can not be defined by measuring viscosity at one rate. One of the most important instruments in the rubber industry is the Sharing Disc Viscometer. A knurled disc within a serrated cavity and measurement of combined effect of shear rate and shear stress are recorded as a function of time. The shear rate is expressed as :

$$y = \frac{r_1 \Omega}{h}$$

and shear stress is expressed as

$$\sigma = \frac{c}{\frac{4\pi r_1^3}{3 + \frac{1}{\eta}} + \frac{4\pi\eta b r_1 h}{1 - \left(\frac{r_1}{r_2}\right)^{2\eta}}}$$

where

r_1	=	rotor radius
r_2	=	stator radius
b	=	rotor thickness
h	=	clearance, top & bottom between rotor and stator
Ω	=	angular velocity of the rotor
c	=	driving torque
η	=	material constant

The combined relation is converted into mooney number with the help of standard torque 84.6 Kg cm = 100 Mooneys.

WORKING :

FF Mooney Viscometer is a shearing disk viscometer, designed for accurate measurement of viscosity, scorch time and cure rates of the elastomers.

The instrument consists of flat, cylindrical disk, driven by a motor to rotate slowly and continuously in one direction. This disk is embedded into elastomer specimen which is confined in a heated die cavity maintained at specified temperature and kept closed by a specified force. As the disk rotates its experiences a shear strain. This resistance to rotation offered by the elastomer is shearing viscosity which is proportional to the mean absolute viscosity of the specimen.

Normally a preheat period is given to the elastomer following which the disk starts to rotate. A initial high viscosity is recorded and then as the viscosity of specimen decreases with time the recorded viscosity also decreases, to a minimum value Viscosity obtained with large rotor are approximately twice those of small rotor.

The viscosity is reported as under:

1. Method of sample preparation
2. Mooney viscosity number
3. Rotor size
4. Preheat time.
5. Time interval to viscosity reading
6. Temperature of test

Typical test results are stated as under :

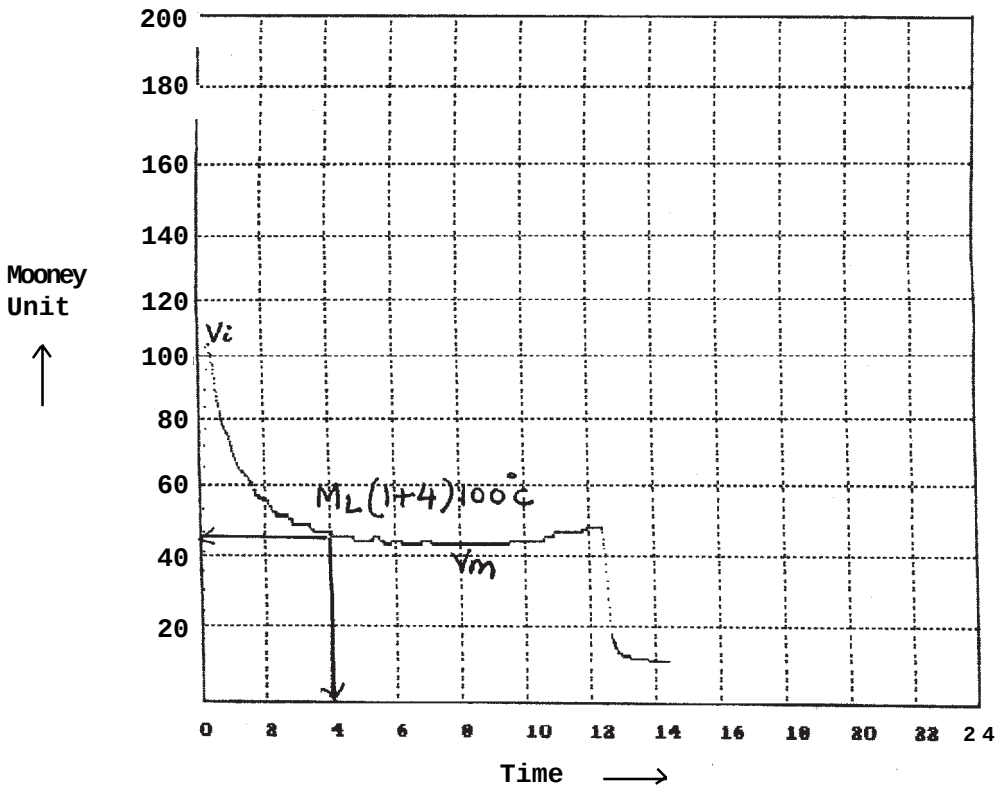
= 50ML(I+4)100°C

where

50 M	=	Viscosity in mooney units.
L	=	Large rotor (for small replace it with ‘S’)
I	=	Preheat time in minutes.
4	=	Time in minutes after starting the motor at which the reading is taken.
100°C	=	Test temperature.

The test temperature and duration of test are specified in international standards

Mooney Graph



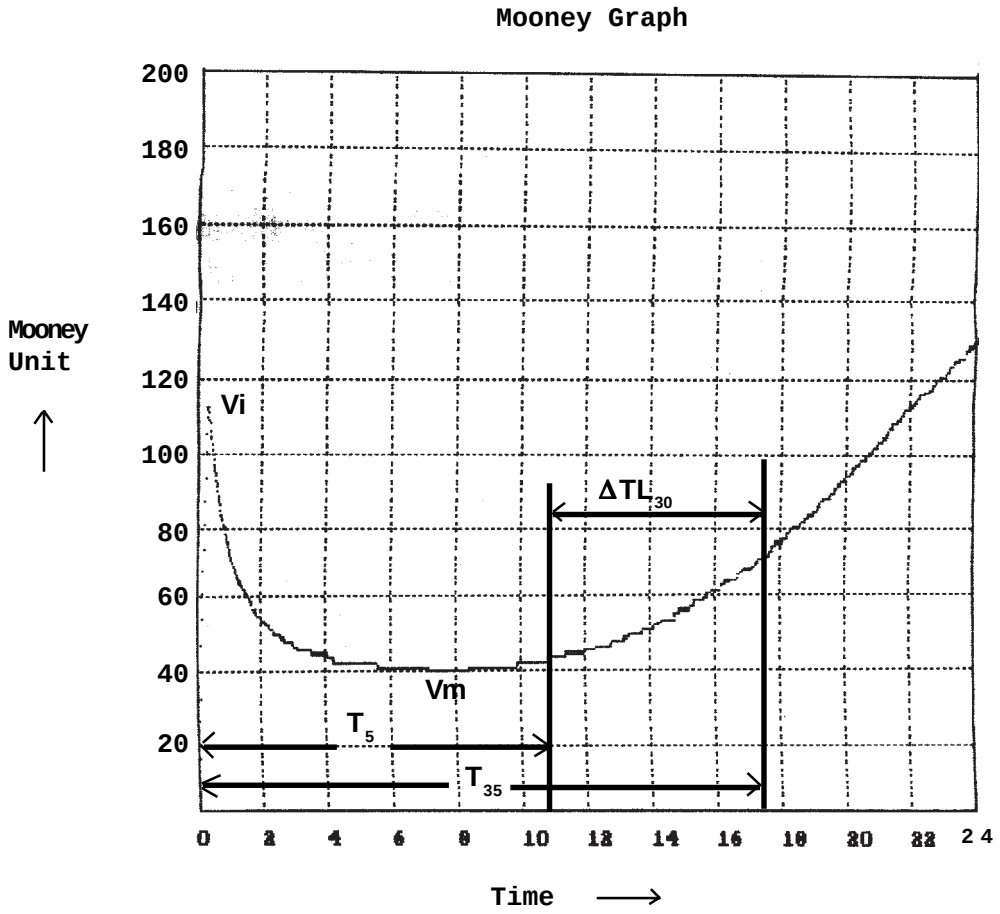
as given in the chart below:

Type of Rubber	Test Temperature (°C)	Running time of rotor (Min.)
NR (Natural Rubber)	100 ± 0.5	4
IIR, BIIR, CIIR	100 ± 0.5 125 ± 0.5 *	8
EPDM, EPM	125 ± 0.5	4
Other Synthetic rubbers black master batches, compounder rubber and reclaimed material	100 ± 0.5	4

* Temperature of 125°C should be used whenever the specimen has viscosity higher than 60ML(I+8)100°C.

MOONEY SCORCH TEST:

Elastomers which vulcanize at the experimental temperature the visocity will increase from minimum value at the induction or scorch point. The rate of increase in viscosity with time is a measure of cure rate of elastomer. This cure characteristics may be determined from plot of viscosity v/s time graph.



The following information is obtained from the plot:

Vi	=	Initial viscosity
Vm	=	Minimum viscosity
T5	=	Time to five point rise from Vm (time to scorch)
T35	=	Time to thirty five point rise from Vm
ΔTL_{30}	=	T35 - T5 Rate of cure or cure Index - for large rotor
ΔTS_{15}	=	T18 - T3 Rate of cure or cure Index for small rotor

SAMPLE PREPARATION :

The Mooney Viscosity is affected by the manner in which the rubber is prepared and the condition of storage prior to test. Accordingly, the prescribed procedure should be followed rigorously as given below in the chart.

The test piece consists of two disks of elastomer of 50mm diameter and approxi-mately 6mm thickness sufficient to fill completely the cavity of the viscometer. The specimen should be free from air and from pockets that may trap air against the rotor and die surfaces. A hole is pierced through the centre of one disk for insertion of rotor stem. The specimen be allowed to rest atleast 30 minutes at standard laboratory temperature before the test and tested not later than 24 hours after homogeni-sation.

Method of sample preparation for Mooney Viscosity test :
(as per ISO 1796)

Type of rubber	Mill roll Temp. (°C)	Nip width (mm)	No. of Passes
NR (Natural Rubber)	70 ± 5	1.3 ± 0.15	10
IIR, BIIR or CIIR	No milling	-	-
BR	35 ± 5	1.4 ± 0.15	10
CR	20 ± 5	0.4 ± 0.05	2
EPDM, EPM	35 ± 5	1.4 ± 0.15	10
Black Master-batch	50 ± 5	1.4 ± 0.15	10
Compounded rubber & reclaimed material	No milling	-	-
Other synthetic rubber	50 ± 5	1.4 ± 0.15	10

When Butyl rubber (IIR, BIIR, CIIR) are tested in crumb form they must be massed as “other synthetic rubber”)

DELTA MOONEY

It is the difference between mooney viscosity at 1.5 and 15 minutes. It reflects the ratio of breakdown to build-up. It is taken as a measure of processibility of the rubber, a higher value being indicative of good processing behaviour.

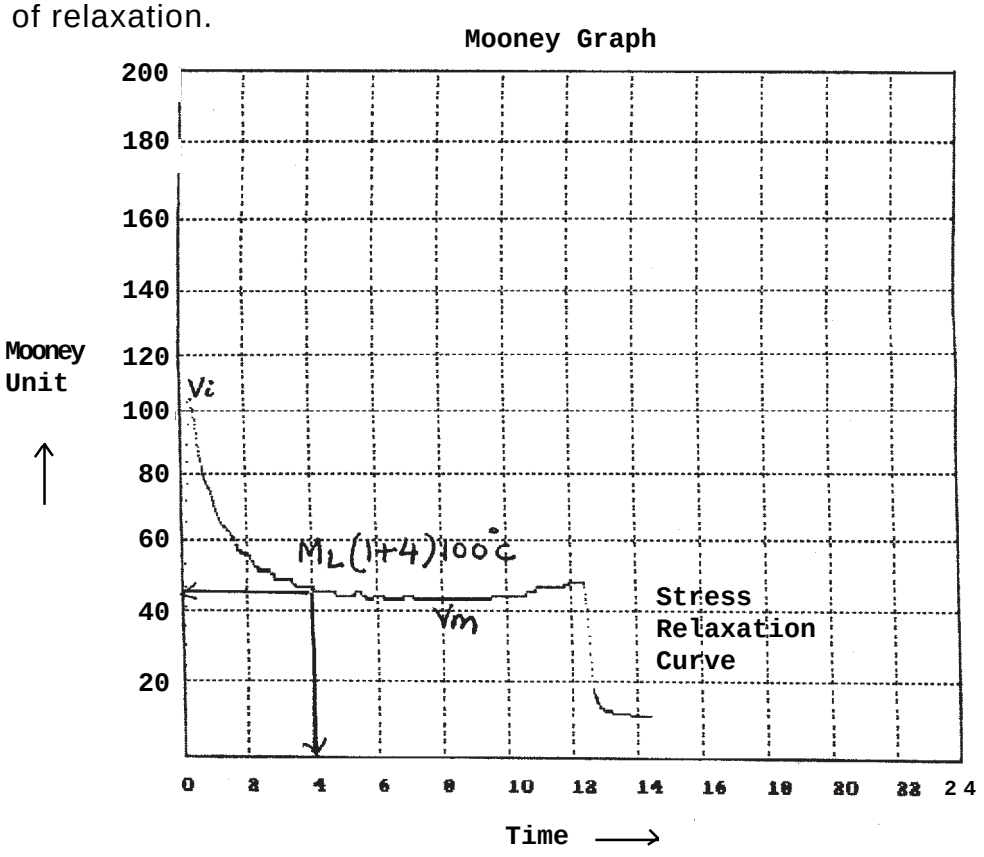
STRESS RELAXATION TEST :

Extension of the basic Mooney viscosity test to include relaxation can generate information on both raw polymer and compounded green stock rheological behavior. the measured stress relaxation data obeys the power law

$$ML = Kt^{-a}$$

where ML is the decaying Mooney stress, t is the relaxation time and K and a are characteristic parameters of the relaxation curve. K is a measure of stiffness of the rubber and is proportional to Mooney viscosity ML 1+4 or ML 1+8. The value "a" is a measure of speed of relaxation, which is a combination of viscous and elastic response of the material (the slope of the log ML vs. log time plot). Parameter "a" correlates with compound processing behavior (milling, extrusion, die swell) and raw polymer molecular weight distribution.

This test helps us to study co-relation between Viscosity and Elasticity of a batch of rubber. It is performed at the end of Mooney Viscosity Test when the rotor is stopped suddenly and the torque is continued to be monitored over a given period of time. This can be calculated as the % torque drop (Mu) achieved after a given period of relaxation.



VISCOSOFT

Viscosoft is a customized software dedicated to “FF Mooney Viscometer”. It has the following major functions.

1. COMPUTER CONTROLLED TESTING: Preheat time and run time duration once entered into the testing specification, the computer automatically gives desired preheat time, starts the test and at the end of test stops the motor.

2. DATA CAPTURE & DISPLAY :

Torque signal generated by the Torque Sensor is appropriately modulated, amplified and then the analogue signal is converted into equivalent digital signal through an efficient Data Acquisition Card. The torque signal is a continuous signal, this is captured, computed upon and displayed on line against time.

3. DISPLAY OF RESULTS :

At the end of the test, the Computer calculates the results automatically and displays it. These results can also be printed as “Run Time Report”

4. DATA CHARACTERISTICS :

Each test specimen qualifies the following seven variables:

a) Specimen Serial No.,
b) Date of testing,
c) Type of test,
d) Preheat time,
e) Temperature of test,
f) Size of Rotor,
g) Condition of specimen, milled/unmilled.

Based on above seven variables, Viscosoft commands to open a Master file (STOCK & CONTROL LIMIT). The computer later on compares and analyses the test data graphically and statistically, based upon these variables.

5. ANALYSIS :

Over a period of time as the data-base builds up, Viscosoft’s “Powerful Analytical Software” analyses it and can present summarized or detailed reports.

6. REPORTS :

a) Run time report: After the completion of test, there is a facility to take out the print report as well as the graph.

b) Stock & Control Limits: Viscosoft offers the option of entering control limits e.g. upper limit,lower limit,related to each parameter monitored during the test. This facility recognises the “FF Mooney Viscometer” as an excellent instrument for comparing each test result against the set limits. The test results which does not fall within the set limits are marked with “Asterisk”. For record purpose, a print of control limits can be taken out.

c) AnalyticalReports: Viscosoft generates the following reports related to period, stock type and status of pass/fail criteria :

i) Statistical Analysis: Based upon search criteria, the data retrieved can be viewed and/or printed under this head. The report can either be detailed or summarised. There is a choice to have a report with or without Graph. The report generated under this head is qualified by the control limits entered in the STOCK AND CONTROL LIMIT head. The results of all the specimen are displayed on a single graph for the viewer to appreciate at a glance.

ii) Graphical Analysis: It is a distinctive facility by which a single parameter from the data retrieved is nicely displayed graphically. The parameter chosen can be any of the following: ML(I+4)100°C, Vm, T5, T35, ΔTL_{30} etc.

ii) Statistical Quality Control (SQC): The facility of SQC has two main advantages:

Firstly, a summary of test reports as well as Statistical analysis of the test results e.g. No. of samples, the upper value, the lower value, mean, range, standard deviation, variance and coef. of variation.

Secondly, the percentage of test results falling within the mean ± 1,2,3, standard deviation can also be known. It helps the Compounder to know about the levels of quality being maintained.

7. CALIBRATION :

Viscosoft performs the calibration such that when a given mass is suspended by the pulley in the machine, the mooney value is displaced. Any deviation from expected value is recorded and incorporated as calibration factor.

8. CONFIGURATION SETUP :

It is basically a display screen which shows various pre-set parameters of the Viscosoft with respect to Viscometer. The users are advised not to change any of these variables, but to seek the service of the Service Engineer for changing/ updating the variables i.e. Configuration Set-up.

9. MAINTENANCE :

“Viscosoft” has a unique facility of updating and re-arranging the data, which can get corrupted whenever the power supply is interrupted.

10. BACKUP & RESTORE :

Viscosoft has the facility to take the back-up of the data on floppy, which can be restored in case of corruption of data or hardware failure.

SPECIMEN TEST-REPORT

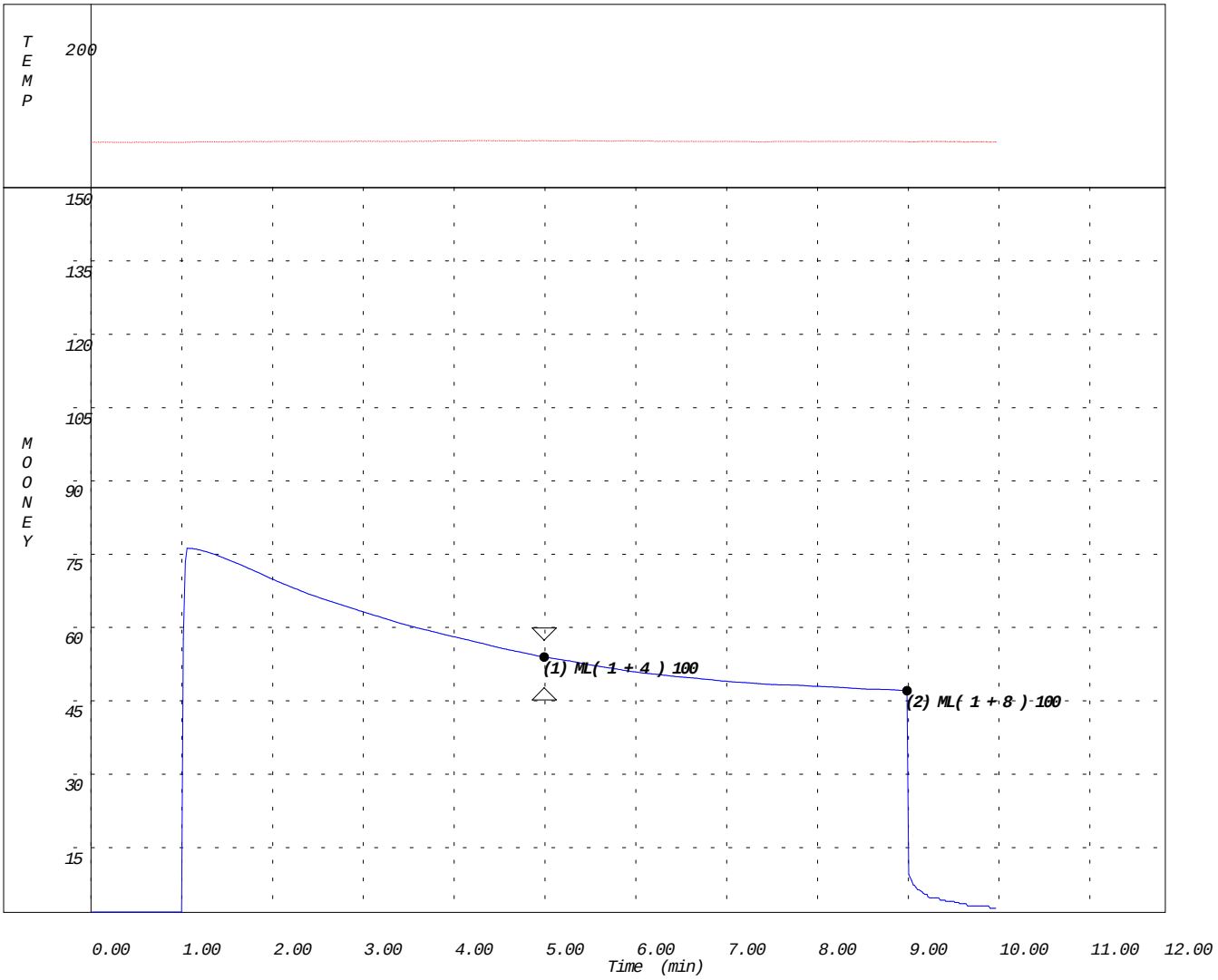
FF-Mooney Viscometer — Page No

Compound Name:	SBR-1502	Stock Mix:	UnMilled	Testtype:	Viscosity Test	Rotor Size:	Large
PreHeat time:	1 min	Temperature :	100 C				
Test Duration:	12min	Mooney Range:	150mu				

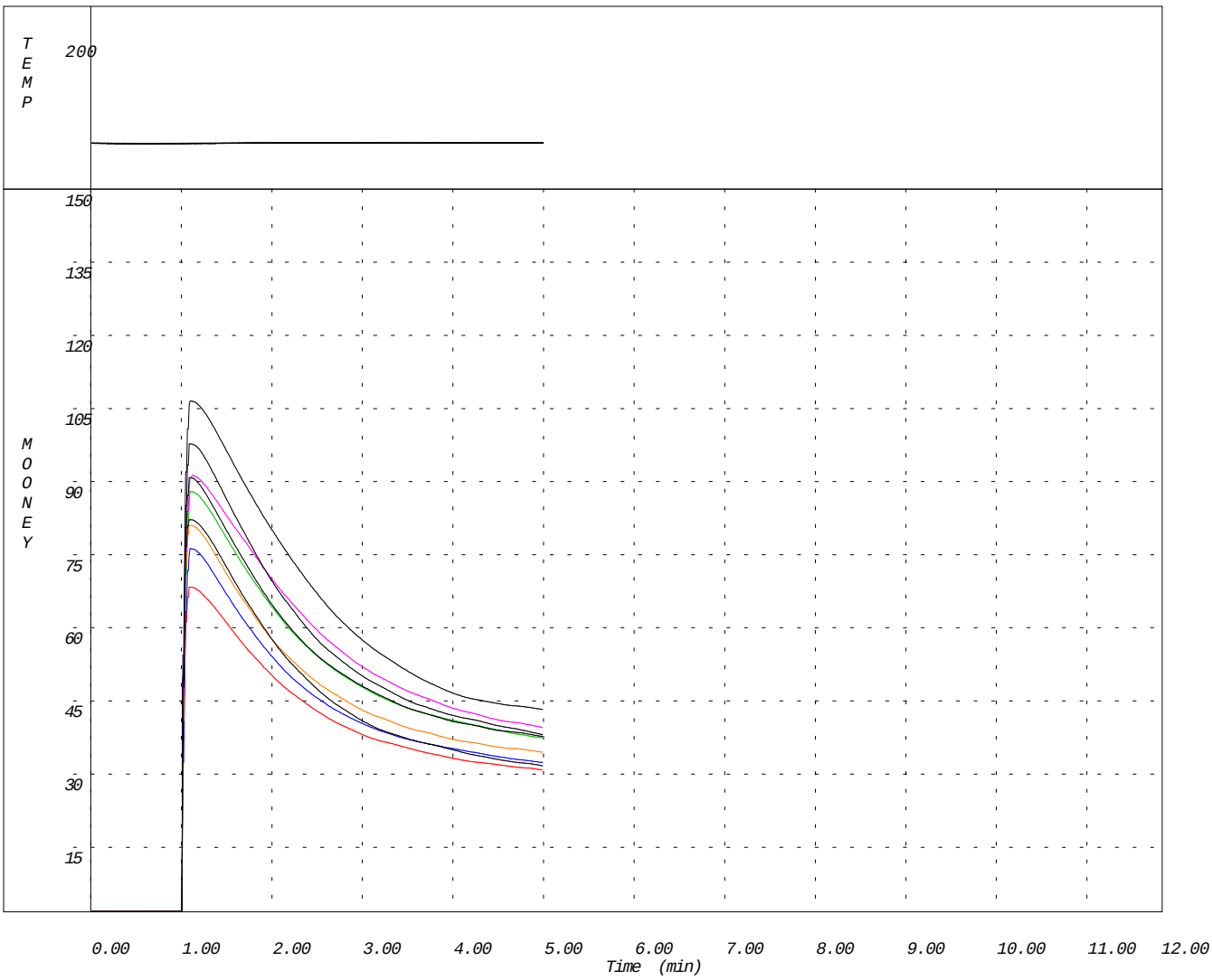
Lower Range:				47	0	0	0
Upper Range:				57	0	0	0

Test No	Batch Id	Test-Date	Init. Mooney	Mini. Mooney	M 4	M 8	M 0	M 0
128	1(1)	24/01/2004	76.25	46.42	53.47	46.42	0	0

FF-Mooney Graph



STATISTICAL ANALYSIS REPORT



STATISTICAL QUALITY CONTROL REPORT

Compound Name: RECLAIM | Stock Mix: UnMilled | Testtype: Viscosity Test | Rotor Size: Large

PreHeat time: 1 min | Temperature : 100 C

Test Duration: 12min | Mooney Range: 150mu

Lower Range:			30	0	0	0
Upper Range:			45	0	0	0

	Init. Mooney	Mini. Mooney	M 4	M 8	M 0	M 0
Total (N)	9	9	9	9	9	9
Range	39.06	13.75	12.72	0	0	0
Mean	87.17	38.6	35.21	0	0	0
Sigma	11.73	4.61	4.21	0	0	0
LSD (5%)	129.8	20.03	16.68	62.7	0	0
Coef. of Variation	13.46	11.94	11.96	110.74	129.11	0
1 Sigma %Reading	78	78	67	100	100	100
2 Sigma %Reading	100	100	100	100	100	100
3 Sigma %Reading	100	100	100	100	100	100

Test No.	Batch	Init. Mooney	Mini. Mooney	M 4	M 8	M 0	M 0
10	1(9)	68.21	32.28	29.76	0	0	0
2	1(1)	88.23	40.26	36.51	0	0	0
3	1(2)	76.26	34.29	31.34	0	0	0
4	1(3)	81.15	36.24	33.5	0	0	0
5	1(4)	91.64	42.85	38.65	0	0	0
6	1(5)	107.27	46.03	42.48	0	0	0
7	1(6)	98.24	41.32	37.19	0	0	0
8	1(7)	91.16	40.03	36.8	0	0	0
9	1(8)	82.37	34.08	30.62	0	0	0

Stress Relaxation Data Analysis Report

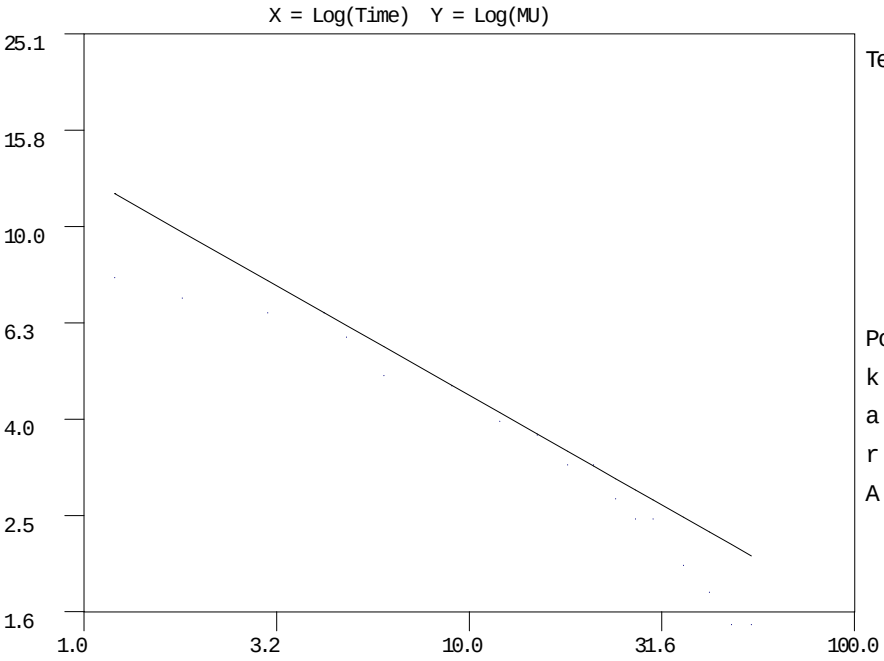
FF-Mooney Viscometer Specimen No: 15 TestDate: 01/10/2003 Page No 1

Compound Name: SBR-1502 Batch Id: 1(1) Operator: BALAJI

Testtype: Viscosity Test Stock Mix: UnMilled Rotor Size: Large

PreHeat time: 1 min Test-Temp: 100 C

Stress Relaxation Test Analysis



Test-Time(sec): 60

Power Law Decay Model

k = 12.7203

a = -0.4551

r = -0.96718

A = 193.9734

Time (sec)	Mooney (MU)
1.20	7.84
1.80	7.11
3.00	6.62
4.20	6.62
4.80	5.89
6.00	4.91
9.00	4.67
12.00	3.94
15.00	3.69
18.00	3.20
21.00	3.20
24.00	2.72
27.00	2.47
30.00	2.47
36.00	1.98
42.00	1.74
48.00	1.49
54.00	1.49

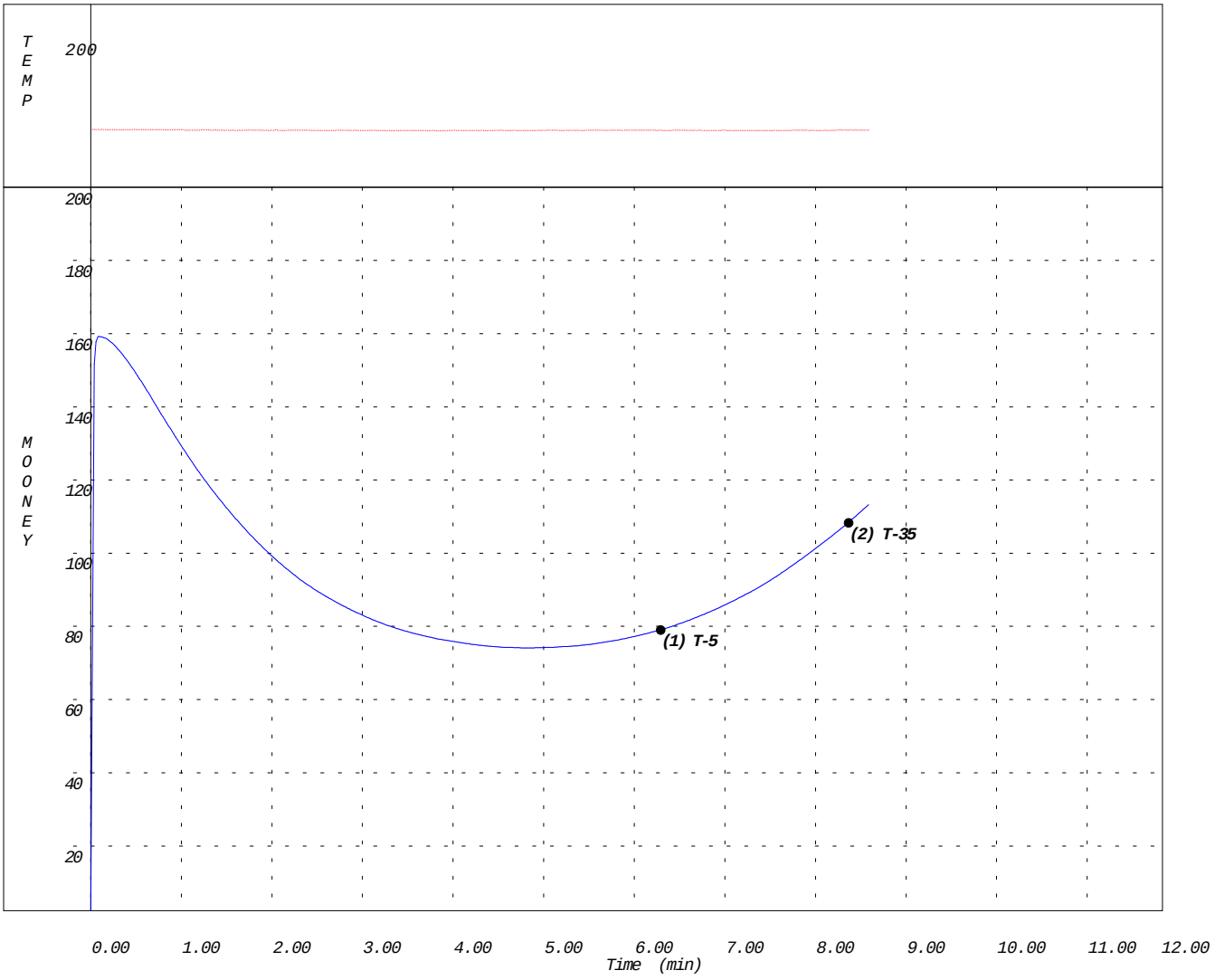
SPECIMEN TEST-REPORT

FF-Mooney Viscometer | Page No

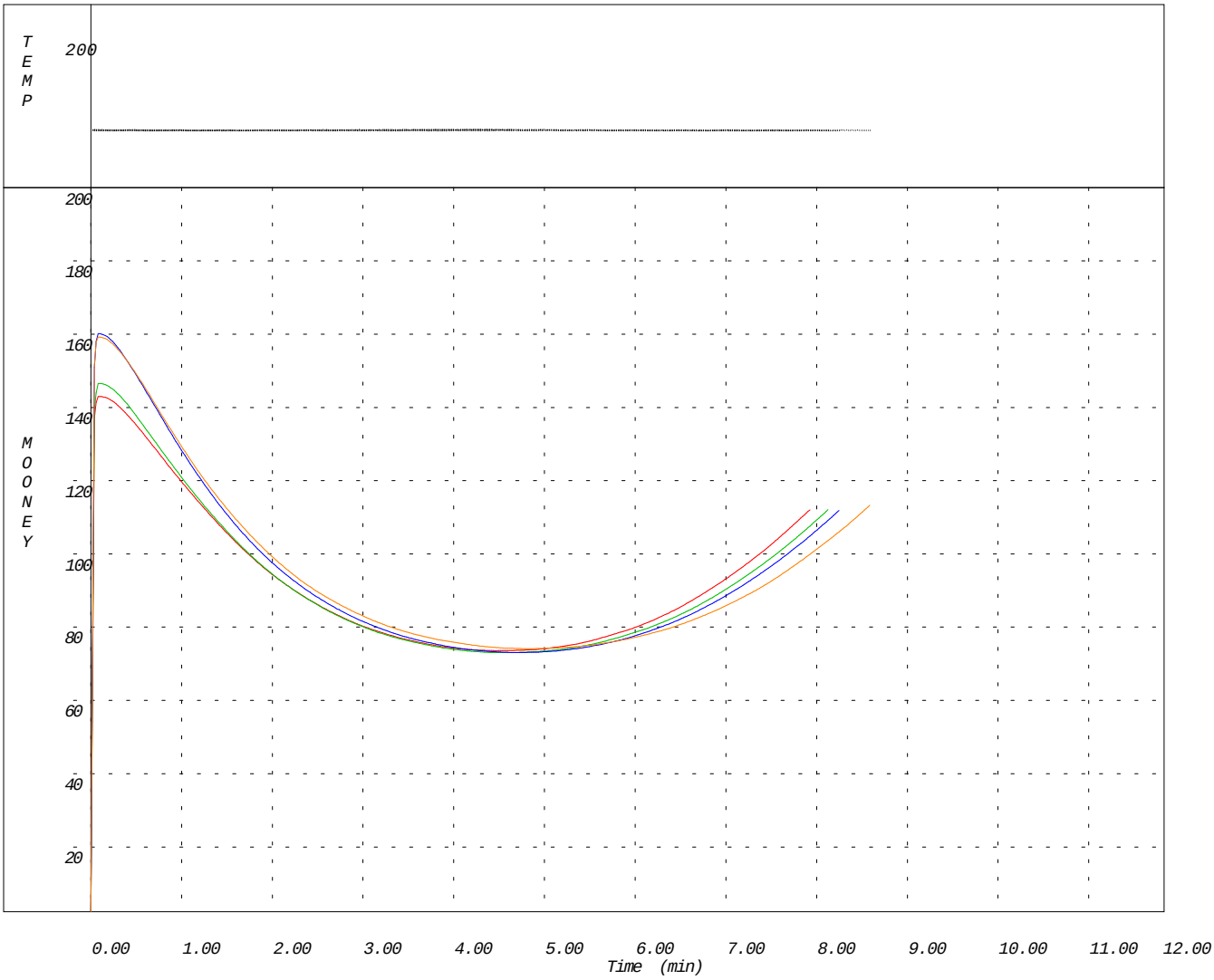
Compound Name:	TEST-S	Stock Mix:	Milled	Testtype:	Scorch Test		Rotor Size:	Large			
PreHeat time:	0 min	Temperature :	121 C								
Test Duration:	12min	Mooney Range:	200mu								
Lower Range:				0	0	0	0	0	0		
Upper Range:				0	0	0	0	0	0		

Test No	Batch Id	Test-Date	Init. Mooney	Mini. Mooney	T-5	T-35	T-0	T-0	T35-T5	T0-T0
183	1(3)	14/02/2004	160.63	73.48	6.3	8.38	0	0	2.08	0

FF-Mooney Graph



STATISTICAL ANALYSIS REPORT



STATISTICAL ANALYSIS REPORT

Compound Name: RECLAIM | Stock Mix: UnMilled | Testtype: Viscosity Test | Rotor Size: Large

PreHeat time: 1 min | Temperature : 100 C

Test Duration: 12min | Mooney Range: 150mu

Lower Range:	30	0
Upper Range:	45	0

	Init. Mooney	Mini. Mooney	M 4	M 8
Total (N)	9	9	9	9
Range	39.06	13.75	12.72	0
Mean	87.17	38.6	35.21	0
Sigma	11.73	4.61	4.21	0
LSD (5%)	129.8	20.03	16.68	0
% Coef. of Variation	13.46	11.94	11.96	0
Cp (Process Capability)			.59	0
Cpk (Process Capability index)			.41	0

Test No.	Batch	Init. Mooney	Mini. Mooney	M 4	M 8
10	1(9)	68.21	32.28	29.76	0
2	1(1)	88.23	40.26	36.51	0
3	1(2)	76.26	34.29	31.34	0
4	1(3)	81.15	36.24	33.5	0
5	1(4)	91.64	42.85	38.65	0
6	1(5)	107.27	46.03	42.48	0
7	1(6)	98.24	41.32	37.19	0
8	1(7)	91.16	40.03	36.8	0
9	1(8)	82.37	34.08	30.62	0

GRAPHICAL ANALYSIS REPORT

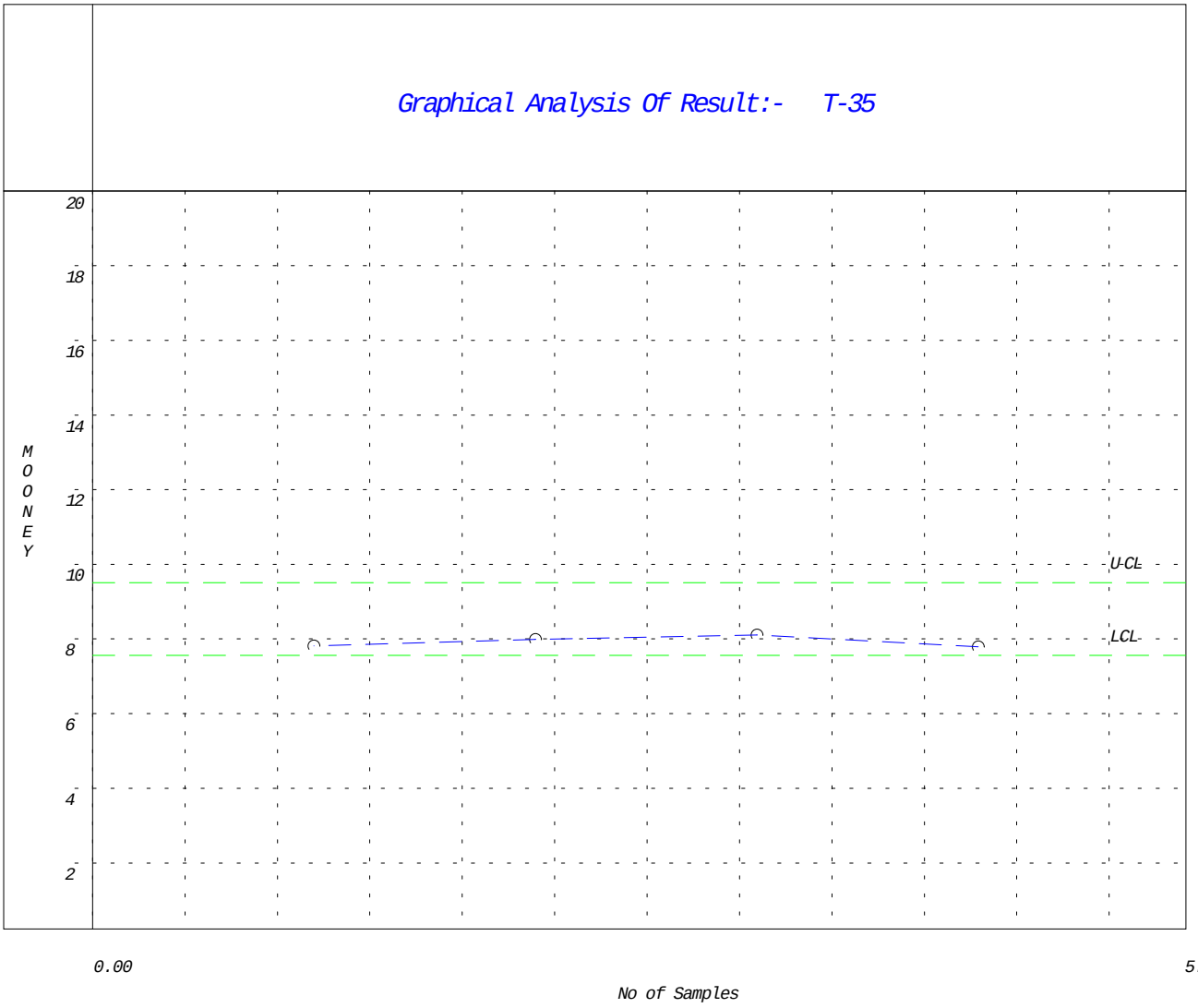
Compound Name:	TEST-S	Stock Mix:	Milled	Testtype:	Scorch Test	Rotor Size:	Large
PreHeat time:	0 min	Temperature :	121 C				
Test Duration:	12min	Mooney Range:	200mu				

Lower Range:	7.5
Upper Range:	9.5

Parameter:- T-35

Total (N)	4
Range	0.32
Mean	7.87
Sigma	0.14
LSD (5%)	0.02
Coef. of Variation	1.77

FF-Graphical Analysis Graph



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