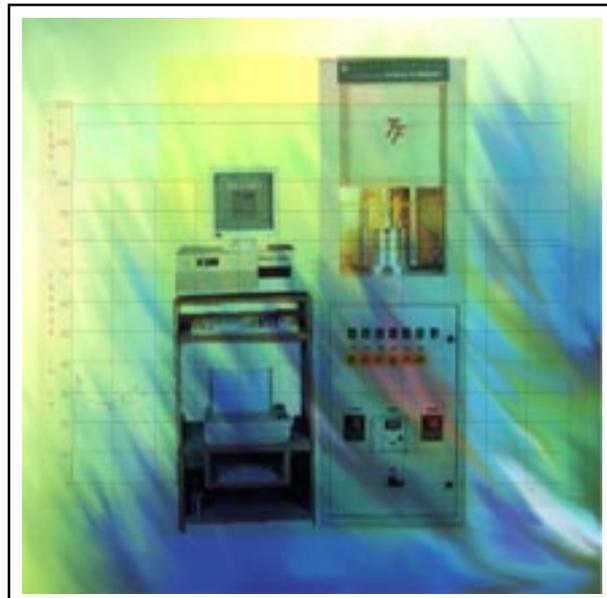


Oscillating Disc Rheometer



SPECIFICATIONS

Oscillation Frequency	:	100 Cycles/minute (1.66 Hz.)
Oscillation Amplitude	:	Plus and Minus 1°, 3°, 5° (Half Cycle).
Sample Volume	:	Approximately 8 cm ³ .
Temperature	:	Microprocessor controlled. Calibrated range 100 - 200° C. Independent Upper & Lower Platen Control Accuracy-control temperature to within ± 0.5°C.
Torque Transducer	:	Directly shaft mounted in line with Oscillating disc. Four arm Temperature compensated strain gage bridge.
Recording & display	:	Directly on-line display on V.G.A. Monitor Memory storage of data. Automatic computation of results. Display of multiple Graphs. Statistical Analysis. Statistical Quality Control.
Printed Data	:	80 column
Electrical Power Supply	:	A.C. 220 V. 50 Hz. 20 Amp. Maximum.
Compressed Air Supply	:	60 psi. (4.2 Kg./Sq. Cm.) minimum operating pressure.
Environment	:	Dust free reasonably controlled ambient temperature and humidity.
Dimensions	:	A. Basic Unit 63 x 26 x 23 Inches B. Computer Unit Desk Top

RHEOMETER

In the Rubber Industry the effects of compound variations on curing characteristics are important in compound development studies or production control. In compound development, the composition of the ingredients can be varied until the desired vulcanization characteristics are achieved. For all this, the Computerized Rheometer with Micro-processor temperature controls is an equipment of vital importance. The Rubber Compounder feels handicapped without a Rheometer. The inventions of new Polymers & Rubber Chemicals leads the compounder to an embarrassing position regarding their choice & use. The Rheometer is an only equipment in the Rubber Industry which helps the Compounder to choose the right material and its appropriate dose to meet the end requirements of the product. The Rheometer not only exhibits the curing characteristics of the Rubber Compound but it also monitors the processing characteristics as well as the physical properties of the material. The "Cure Curve" obtained with a Rheometer is a finger print of the compound's vulcanization and processing character. In fact, the Rheometer has manifold advantages e.g. :

i) RESEARCH & DEVELOPMENT: The most tedious part in compounding is to develop a new compound. 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 Rheometer, one can do all this exercise quickly with minimum wastage of materials.

ii) 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 Rheometric analysis, one could in a large sample size, workout upper and lower control limits, range, mean and standard deviation, with reference to Rheological 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 Rheometer viz. "Rheosoft" is ideal in this regard. By this method, one can monitor easily consistency of each mix every day.

iii) PROCESS CONTROL: The Rheometer gives us the true picture regarding the processing behavior of the rubber compound. By having the complete picture i.e. Viscosity, Scorch time, Optimum cure time of rubber compounds, the compounds are stored, processed and used accordingly. Unused compounds left for longer periods will tend to cure rendering it unsuitable for future use. Such mixes can be tested easily on the Rheometer and decision can be taken regarding their utility. The ability of a Rheometer to detect minor changes in the composition of Rubber Compound has made it a widely accepted Production Control Test Equipment.

iv) EFFECT OF NEW INGREDIENTS: The Rheometer is useful to carry out the study of any of Rubber or Chemicals changed in the existing compounds. The effect caused due to change in the ingredients can be observed on Rheometric Curve.

v) OPTIMIZATION OF INGREDIENTS DOSE: Whenever, the need to change the dose of any of the Ingredients in the compound is felt, its effect can be noticed straight away from the Rheo Curve and accordingly the change can be effected.

vi) ASSESSMENT OF PHYSICAL PROPERTIES: In addition to processing and curing characteristics, the physical properties of the Rubber Compound can also be assessed by experience.

vii) ECONOMICS: Everything said, but one may still be hesitant in investing in such instruments mainly because of its cost. Experience has shown that if Rheometer's full potential is exploited with regards to few of various advantages mentioned above, the return is quite handsome. Improved quality, minimized wastage, optimized dosage of each ingredient, choice of right ingredients, controlled process certainly makes this instrument singularly ideal for any Rubber Industry. In other words one can say Rheometer is "**Compounder and Quality Managers delight**".

OSCILLATING DISC RHEOMETER

PRINCIPLE :

The oscillating Disc type Rheometer is an efficient, simple and reliable testing equipment. It is quite easy to operate. The Rheometer describes precisely and quickly curing & processing characteristics of vulcanizable rubber compounds. It works on a very simple principle.

A test piece of rubber compound is contained in a sealed test cavity under positive pressure and maintained at a specified elevated temperature. A Rotor (biconical disc) is embedded in the test piece and is oscillated through a small specified rotary amplitude. This action exerts a shear strain on the test piece and the torque (force) required to oscillate the disc depends upon stiffness (shear modulus) of the rubber compound. The stiffness of the specimen compound increases when crosslinks are formed during cure. The direct proportionality posited between the shear modulus and the cross linking density is based on the statistical theory of rubber elasticity (cf. Nitzsche/wolf: struktur und physikalisches verhalten von kunststoffen, Berlin, Heidelberg, Vienna: Springer - Verlag, 1962, pp.234ff).

It gives the equation:

$$G = v.R.T.$$

where

G is shear modulus, in n/MM²

v is cross linking density, in Mol/MM³.

R is the universal gas constant, 8.313 J/(Mol.k)

T is the thermodynamic temperature, in K

If strain is directly proportional to the force applied then:

$$\tau = G.y = v.R.T.y$$

where

y is shear strain, equal to the tangent of the shear angle.

τ is the shear stress in n/MM².

if T and y are constant, then τ is proportional to v.

At a constant temperature of the test, a cross linking isotherm is the function of time of that property which serves to measure the course of the cross linking reaction. In the context of Rheometer, the cross linking isotherm is thus the function of time, of the oscillating shear force F, or of the Rheometer indication proportional to it, occurring at a given temperature as a result of vulcanization and expressed as $F = f(t)$

where t is vulcanization time.

A complete “Cure Curve” is obtained when the recorded torque value either increases to an equilibrium value or a maximum value. The time required to obtain a “Cure Curve” is a function of the test temperature and the vulcanization characteristics of the Rubber compound specimen.

The signal of torque (force) is sensed by a torque sensor mounted directly on the torque shaft bearing the rotor under stress. Thus the torque is read directly. This torque signal is converted to volts and then through ADC fed into the computer to draw torque against time curve called “Rheograph”. The following measurements are automatically computed by the computer :

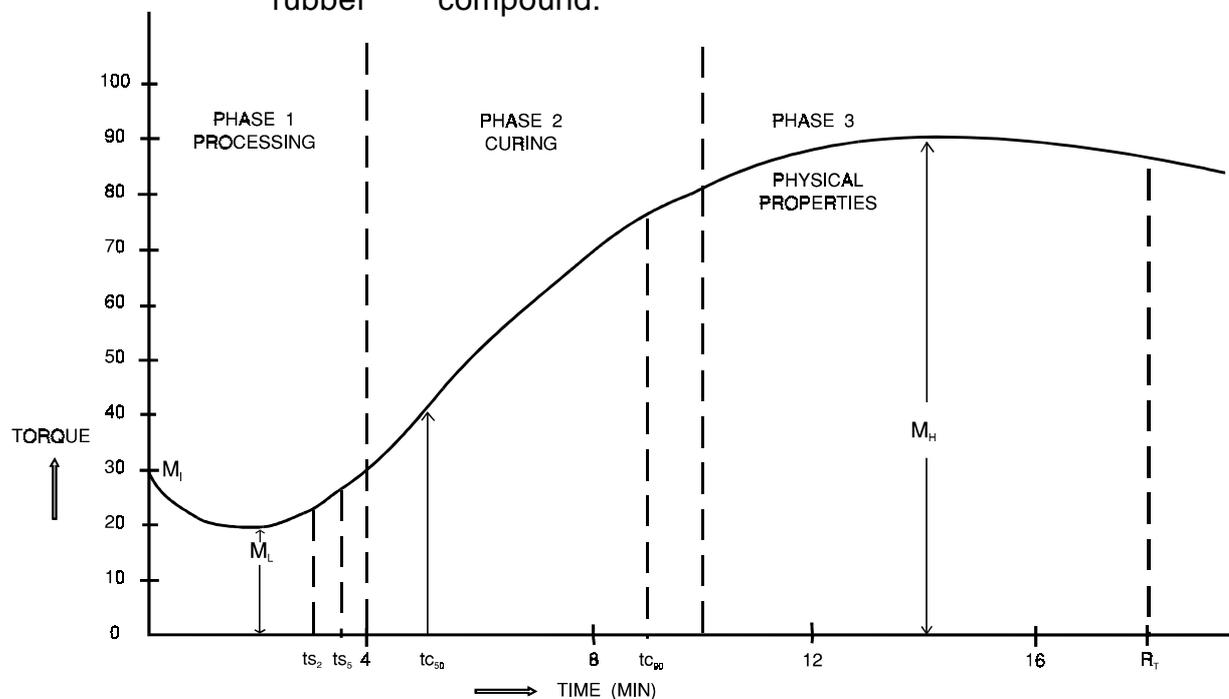
- i) Torque Values : MI, ML, MH/MHR
- ii) Time Values : ts2, ts5, tc50, tc90
- iii) Derived Values : Thermoplasticity, Cure rate,
Reversion time

The minimum torque is proportioned to the viscosity of the uncured compound. The scorch time is a measure of process safety. The full curved torque is a measure of shear modulus or stiffness of the compound.

RHEOGRAPH

Figure 1 shows a typical “Cure Curve” obtained with a “FF Oscillating Disc Rheometer”. From this curve of Torque Vs Cure Time, all the vulcanization characteristics of the Rubber Compound can be determined directly. XY Plot of Torque (force) against real cure time is called “Rheograph”. Rheograph is divided into 3 Phases :

- A) Phase - 1. It gives an indication of processing behavior of the rubber compound.
- B) Phase - 2. It describes the curing characteristics of the rubber compound.
- C) Phase - 3. It gives good indication of physical properties of the rubber compound.



TYPICAL RHEOMETRIC CURVE

The plot of torque against time is analysed to obtain the various results. In “FF Oscillating Disc Rheometer”, The Rheograph is displayed in real time and at the end of test time, computer analyses the graph and results are automatically computed and displayed on the screen. Displayed results are categorized into three columns:

1. Torque values with units lbin.
2. Time values with unit minutes
3. Derived Values.

The significance of these displayed values are:

1. TORQUE VALUES:

- i) MI (Initial Torque). It is the torque recorded at time zero at the start of the test.
- ii) ML (Minimum Torque): As the compound gets heated under pressure, the viscosity decreases and the torque falls. The lowest value of Torque recorded is called ML. Basically, it is a measure of the stiffness and viscosity of unvulcanized compound.
- iii) MH (Maximum Torque): As the curing starts, the torque increases proportionately. Depending upon the type of compound, the slope of rising torque varies. After a while the torque typically attains maximum value and it plateaus out. It is called "Plateau Curve". If test is continued for sufficient time, the reversion of cure occurs and torque tends to fall. This type of curve with reversion is called "Reverting Curve". At times the torque shows continuous rising trend during the period of record. Such type of curve is called "Rising or Marching Curve". MH (Max. torque) is the highest torque recorded in plateau curve. In reverting curve, the Max. torque recorded is abbreviated as MHR.

2. TIME VALUE:

- i) ts_2 (Induction time). After attaining minimum torque, during cure phase, as the torque rises, ts_2 is scorch time for viscosity to rise 2 units above ML.
- ii) ts_5 (Scorch time). It is the time for viscosity (torque) to rise 5 units above ML. Both ts_2 and ts_5 are measures of initial slope of curing phase of Rheograph i.e. these are measures of processing safety.

Scorch is premature vulcanization in which the stock becomes partly vulcanized before the product is in its final form and ready for vulcanization. It reduces the plastic properties of the compound so that it can no longer be processed. Scorching is the result of both the temperatures reached during processing and the amount of time the compound is exposed to elevated temperatures. This period before vulcanization starts is generally referred to as "Scorch time". Since scorching ruins the stock, it is important that vulcanization does not start until processing is complete.

- iii) tc_{50} (Optimum cure time). It is the time at which 50% of cure has taken place.
- iv) tc_{90} (Optimum Cure time). It is the time at which 90% of cure has taken place.

3. DERIVED VALUES :

i) Cure Rate : $CR = 100/(tc_{90}-ts_2)$

The cure rate is an essentially a measure of the linear slope of the Rising Curve.

The rate of cure is the rate at which cross-linking and the development of stiffness (Modulus) of the compound occur after the scorch point. As the compound is heated beyond the scorch point, the properties of the compound changes from a soft plastic to a tough elastic material required for use. During the curing phase cross links are introduced which connect the long polymer chains of the rubber together. As more cross links are introduced, the polymer chains become more firmly connected and the stiffness (modulus) of the compound increases. The rate of cure is an important vulcanization parameter since it determines the time the compound must be cured i.e. the cure time.

ii) Thermoplasticity : $T_p = (MI-ML)$

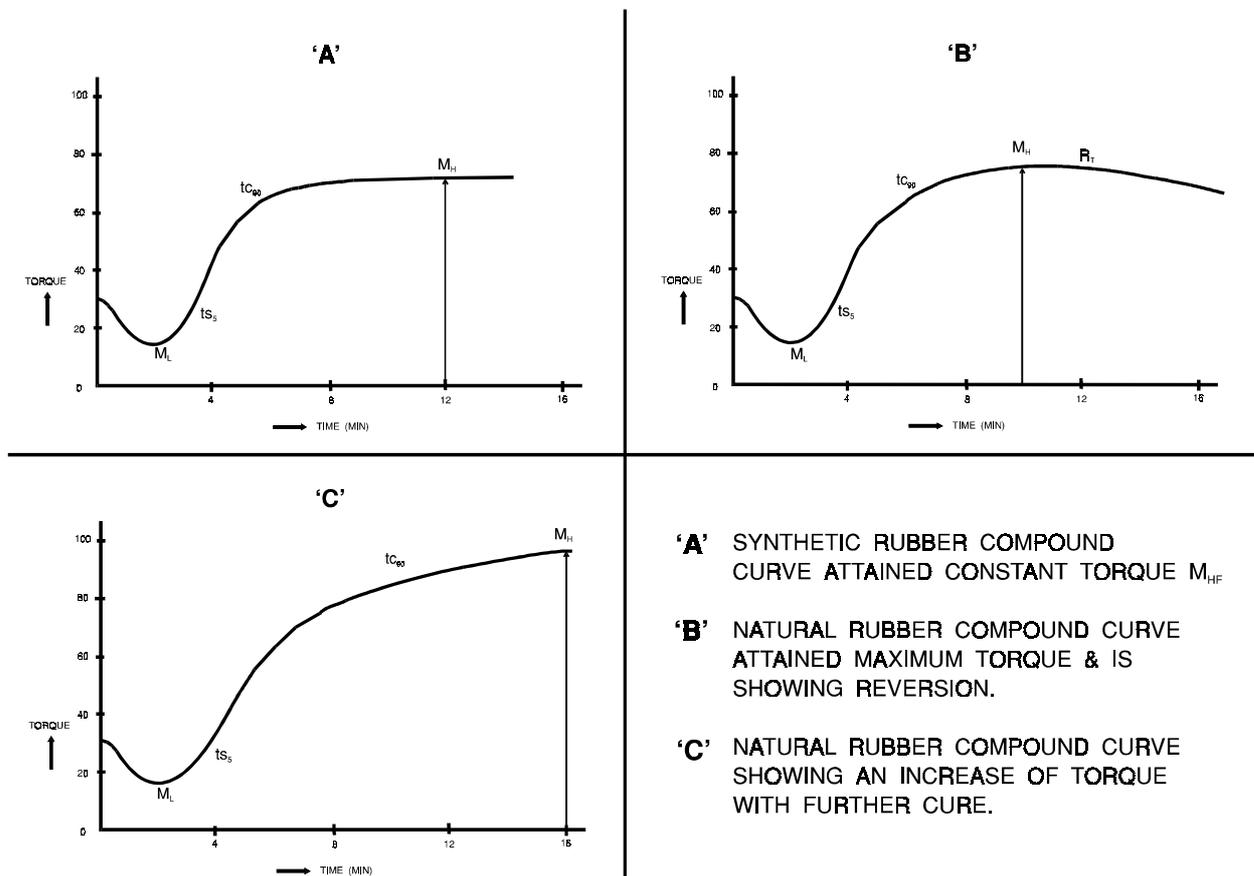
The Thermoplasticity is derived from the difference of initial viscosity & minimum viscosity.

iii) Reversion time (RT) : It is the time to reach 98% MH after passing MH. The reversion time is recorded in minutes. It gives us an indication of the quality of the compound as to how long it retains its physical properties when subjected to heat ageing. Reversion occurs with over cure and the Modulus & Tensile Strength decreases.

INTERPRETATION AND APPLICATIONS OF RHEOGRAPH

The “FF Oscillating Disc Rheometer” produces a Rheograph which has all the three phases with a characteristic shape. A trained eye can monitor the initial trough i.e. processing characteristics of the compound, the slope of rise during curing phase i.e. the curing characteristics of the compound and the further shape of Curve i.e. the anticipated physical properties of the compound.

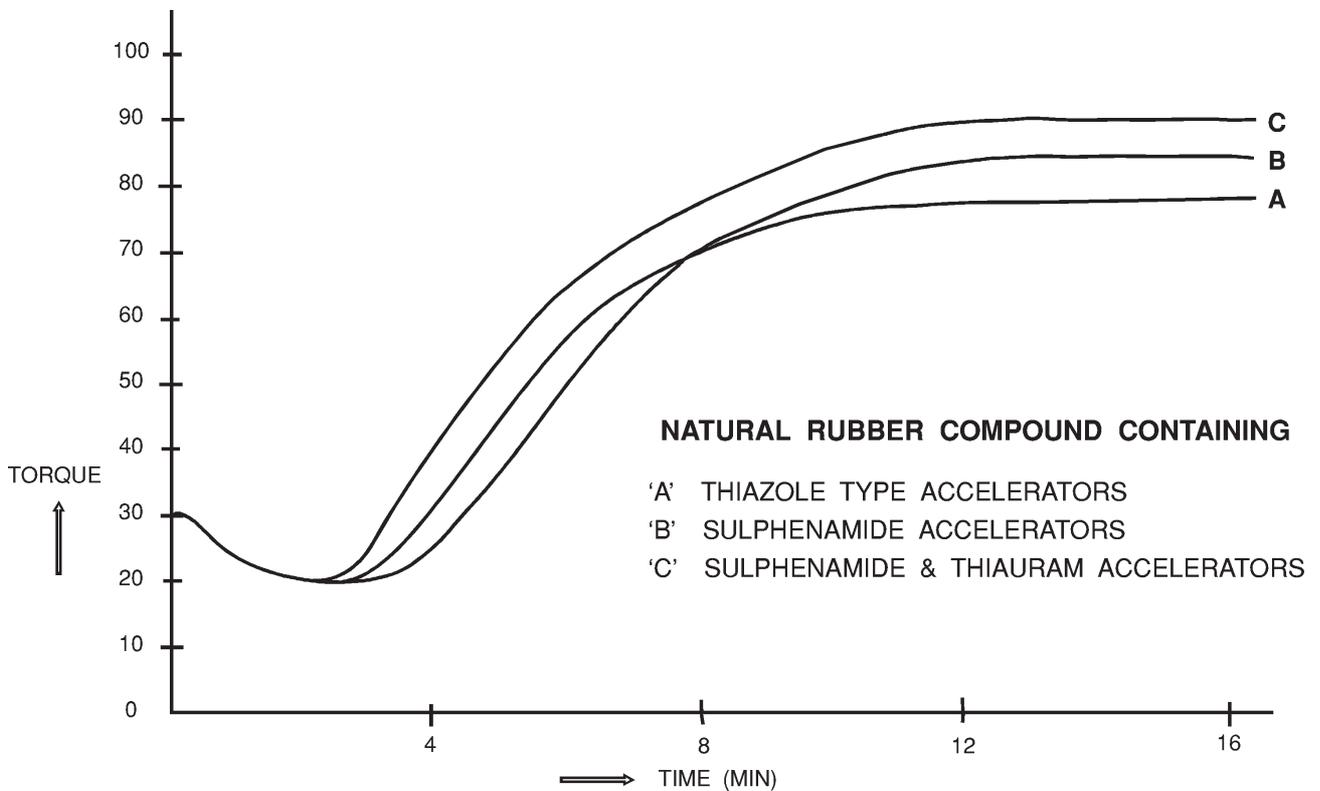
- i) Figure 2 shows the three different types of “Cure Curves” which are obtained with different types of rubber compounds. Curve-1 is of Synthetic Rubber Compound which has attained a constant torque M_H . Curve-2 is of Natural Rubber Compound which has attained the maximum torque and is reverting MHR. Curve-3 is also of Natural Rubber Compound which is showing an increase of torque with further cure. In this case, the compound is continuing to harden, the modulus is rising and the tensile strength as well as the elongation continues to drop.



(Fig. 2)

The rubber compounder normally strives to design and develop a compound which neither reverts nor increase in modulus with overcure.

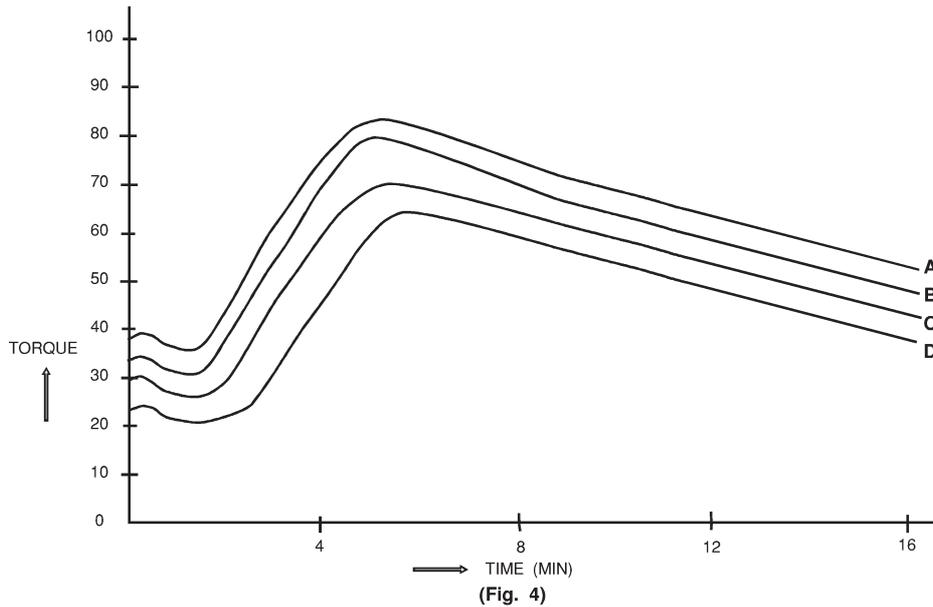
ii) Figure 3 shows the behavior of various types of accelerators in a Natural Rubber compound. The Sulphenamide accelerators behave faster than thiazole type accelerators at vulcanizing temperatures. Thiauram type accelerators acts as a booster when used in combination with Sulphenamide and thiazole type accelerators.



(Fig. 3)

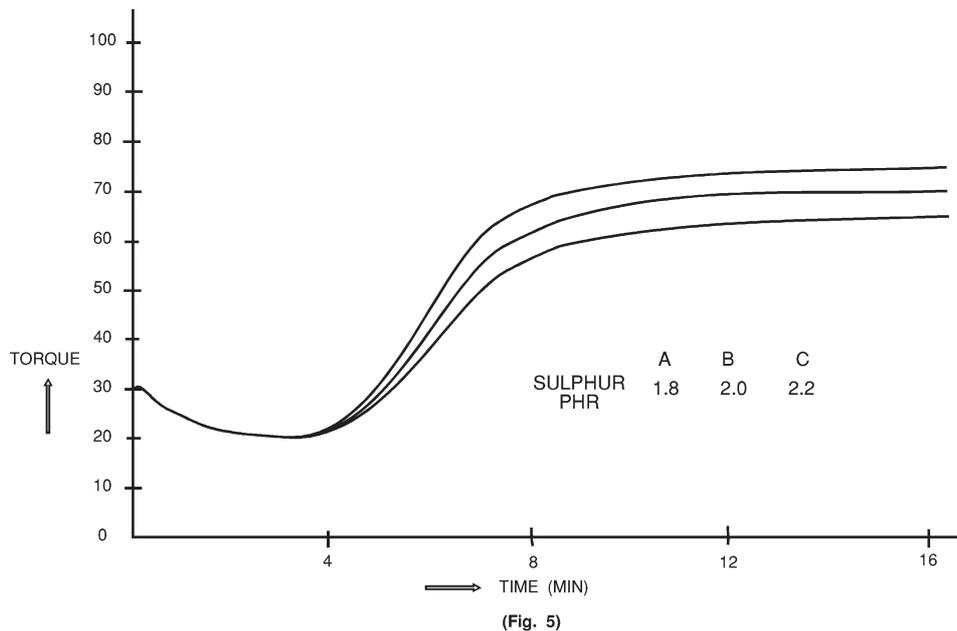
RHEOGRAPH WITH NATURAL RUBBER COMPOUND & DIFFERENT ACCELERATORS

iii) The effect of any of the ingredients whenever changed in the compound can also be observed from the Rheograph. Figure 4 represents a typical NR Compound with variation in oil dosage in the compound.



A TYPICAL NR COMPOUND SHOWING EFFECT OF OIL VARIATION IN REVERTING TYPE CURVES
 OIL A=0, B=2, C=5, D=10

iv) Figure 5 shows how relatively minor changes in the concentration of a compounding ingredient e.g. "Sulphur" changes the characteristics of the "Cure Curve".



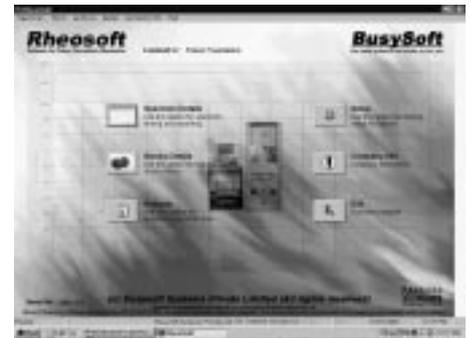
RHEO CURVES OBTAINED WITH MINOR CHANGE IN CONCENTRATION OF SULPHUR IN SBR COMPOUND

Rheosoft features and functions

Easy operations

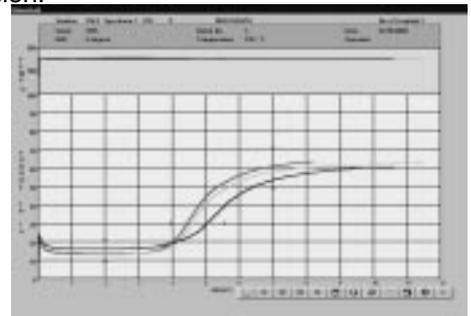
User has to perform three easy steps:

- Fill stock details to keep master parameters of the stocks being tested
- Fill specimen details and run machine testing
- Analyzing the sample test results



Advance specimen testing options

- Continuous testing of samples depending upon batches, stock and session.
- Automatic machine start & stop for each specimen
- User can override automatic function to manual
- Quick loading of specimen for large number of testing
- Each specimen graph is shown with separate color.
- The user can also define this color scheme of graphs.
- The user can see one or many graphs in the session while testing.



Keeps Complete information of Specimen tested

- Stock compound name
- Operator
- Batch
- Session
- Date and time of testing
- Specimen results
- Pass fail results
- QC results

Advance Specimen testing results

- Results are calculated automatically on completion and shown on screen
- Quick redisplay of testing graph
- User can add own comments for future reference for each specimen
- Can add modify and view stock details during specimen testing
- Can view QC of tested specimen
- Can recalculate QC parameters of tested specimen after changing QC criteria
- Query option for finding and listing specimen details
- A graphical report can also be seen on screen while testing to compare the specimen

Stock details and controls

- Compound identification
- Testing temperature
- Torque arc
- Run duration
- Sets control parameters
- Quality control gates
- Pass fail criteria

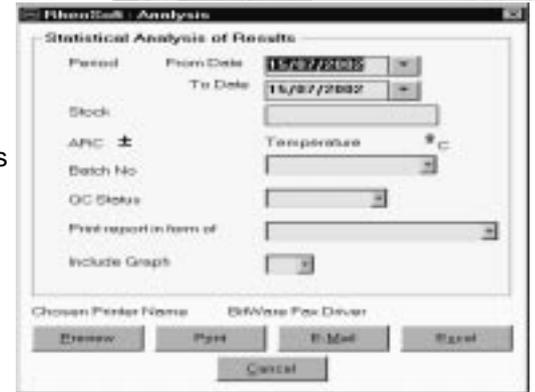
■ Analysis

- User can compare the results using graphical and statistical analysis
- The statistical analysis can also help in setting control parameters of stocks
- Analysis reports are
 - ◆ Specimen runtime report
 - ◆ Statistical analysis report
 - ◆ Graphical analysis report
 - ◆ Statistical Quality Control report



■ Flexible Printing options

- User can select Period of reports, Stock, batch number, QC status
- The reports can be printed with graph without graph or both
- The reports can be sent to
 - ◆ Printer
 - ◆ Email
 - ◆ Fax
 - ◆ MS Excel files



■ User Security

- Each user is identified and can be given a user id and password
- All activities performed by the user are marked against that user ID
- This helps in controlling unwanted activities in the system.
- Users can be allowed to do one or many functions of the software



■ User setups

- Configuration setup of graphs parameters
- Calibration of machine
- Report heading and printer setup parameters
- Graph background and foreground colour parameters
- Change user password
- Email and fax setup



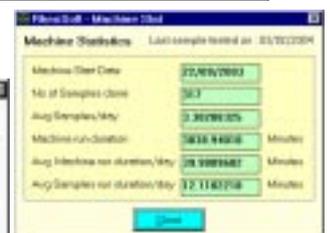
■ Data Base Maintenance

- The software provides automatic or user defined data backup option
- The user can define the days after which data backup is taken
- The user can define the days after which data is compacted
- The user can copy, delete specimen for a period



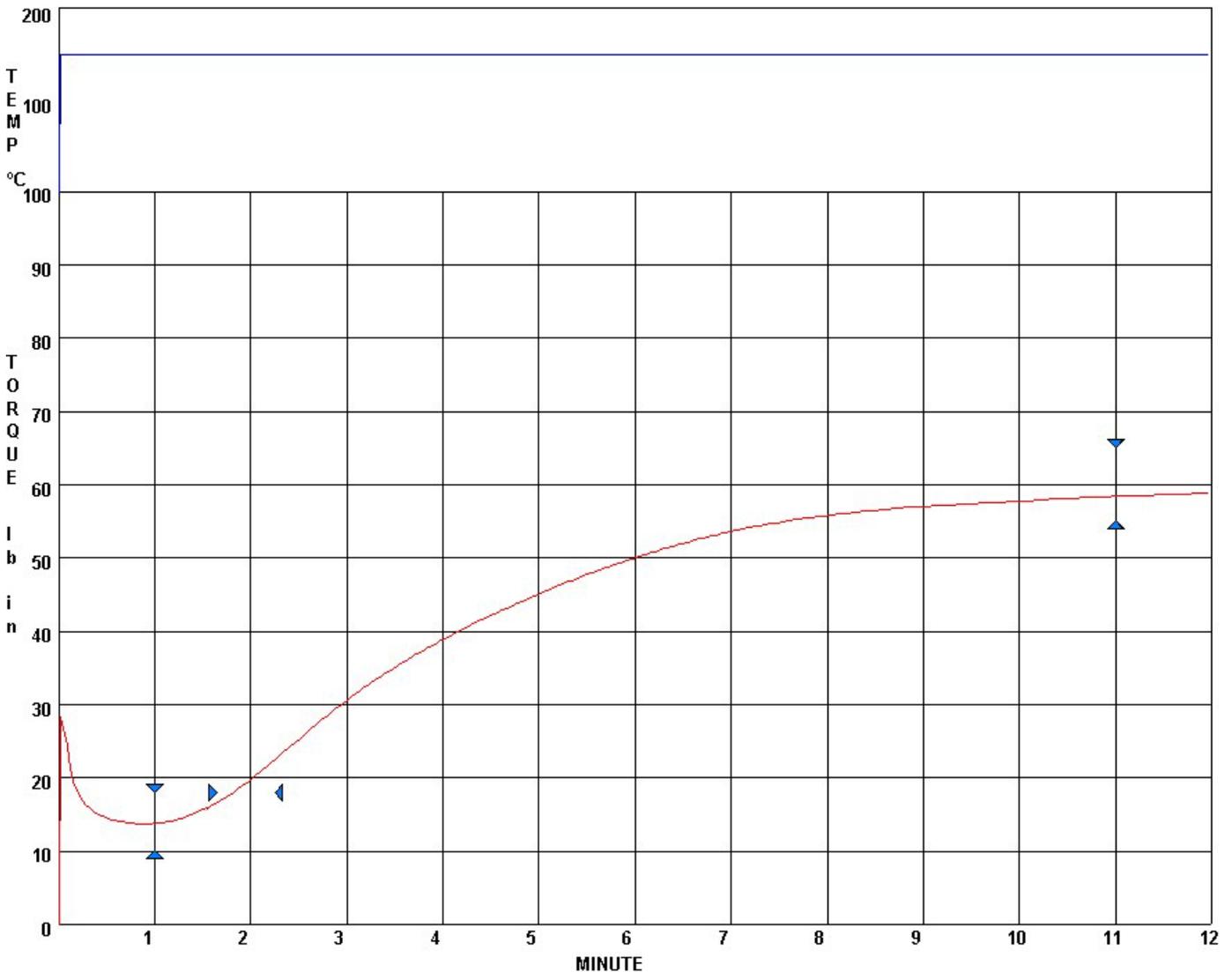
■ Machine running & maintenance record

- Software keeps record of Machine usage in minutes
- Software keeps record of
 - ◆ Total samples done
 - ◆ Average samples/day done
 - ◆ Machine run duration
 - ◆ Average Machine run duration/day
 - ◆ Average sample run duration/day



RUN TIME REPORT

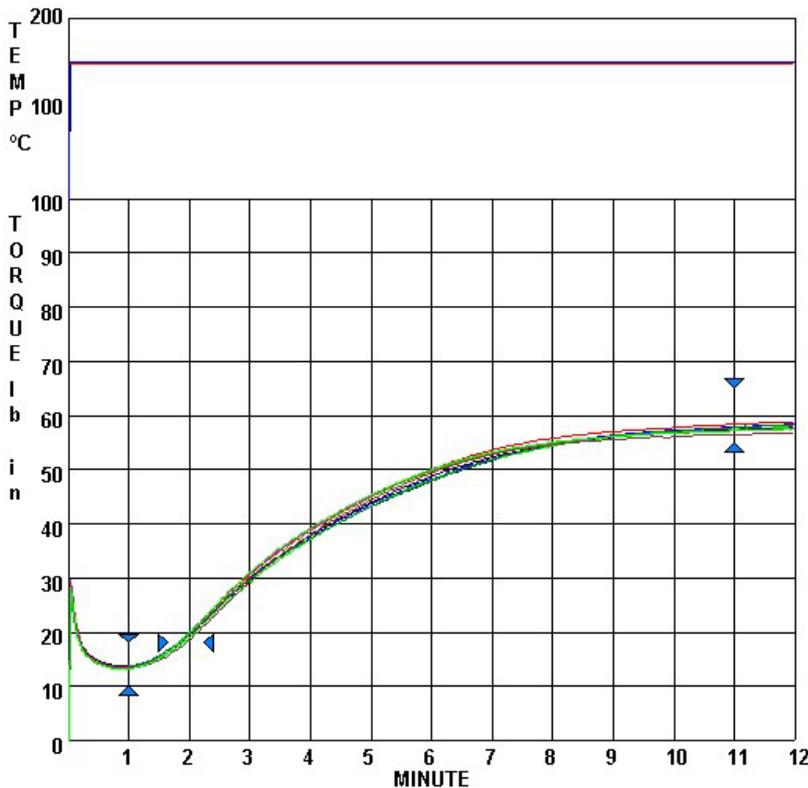
RHEO-SOFT		Session 77		Date 31/12/2003		Page No: 1	
Specimen #	219	Stock	TEST	Batch No.	1		
Date	16/12/2003	ARC	3 Degree	Temperature	150 °C		
Torque Values (lb in)		Time Values (Min)		Derived Values			
MI Initial Torque	28.255	Tb Stop Time	11.97	Thermoplasticity	14.540		
ML Minimum Torque	13.715	T Minimum	.83	Cure Rate(CR)	17.46		
MH Maximum Torque	58.854	T Maximum	11.97	Reversion Time(RT)	.00		
Optimum Cure	54.340	TS2 Induct Time	1.54				
		TS5 Scroch Time	1.90				
		TC'(35) Time	2.89				
		TC'(50) Time	3.67	Trend	Marching		
		TC'(90) Time	7.27	End Temp	149.700		



STATISTICAL ANALYSIS OF RESULTS

RHEO-SOFT		From - 16/12/2003		To - 16/12/2003		DETAILED		31/12/2003		Page No. 1						
Stock : TEST		ARC± : 3 degree						Temperature : 150.00 °C								
Run Duration : 12 Min. (X-axis-Time)								Torque Range : 100 lb in								
Upper Value	35.000	18.000	65.000	1.75	2.55	4.00	8.85	18.00	20.00	18.000	2.25	65.000				
Lower Value	25.000	10.000	55.000	1.25	1.85	3.20	6.85	12.00	14.00	10.000	1.65	55.000				
Mean Value	30.000	14.000	60.000	1.50	2.20	3.62	7.85	15.00	17.00							
Sigma Value	.000	.000	.000	.00	.00	.00	.00	.00	.00							
Date	Spec. Srl No.	Batch	Initial Torque	Minimum Torque	Maximum Torque	ts2	ts5	tc50	tc90	TP	CR	QC1 Torque	QC2 Time	QC3 Torque	End Temp	TR R
16/12/2003	219	1	28.255	13.715	58.854	1.54	1.90	3.67	7.27	14.54	17.46	13.787	1.80	58.418	149.70	M P
16/12/2003	221	2	28.458	13.566	58.230	1.52	1.91	3.79	7.74	14.89	16.08	13.775	1.84	57.772	151.15	M P
16/12/2003	222	3	27.726	13.620	58.366	1.52	1.91	3.77	7.69	14.11	16.22	13.709	1.85	57.814	150.60	M P
16/12/2003	223	4	29.638	13.715	57.619	1.54	1.90	3.57	7.17	15.92	17.77	13.744	1.83	57.169	150.30	M P
16/12/2003	224	5	27.767	13.580	57.850	1.59	1.98	3.82	7.65	14.19	16.49	13.614	1.88	57.404	151.25	P P
16/12/2003	225	6	28.702	13.729	57.714	1.58	1.94	3.71	7.41	14.97	17.15	13.729	1.84	57.414	149.40	P P
16/12/2003	227	8	26.424	13.634	58.148	1.51	1.89	3.79	7.65	12.79	16.28	13.724	1.80	57.731	150.55	M P
16/12/2003	229	1	27.279	13.322	57.606	1.53	1.89	3.58	7.12	13.96	17.89	13.408	1.85	57.172	149.80	P P
16/12/2003	230	2	29.028	13.444	56.793	1.64	2.00	3.64	6.89	15.58	19.07	13.444	1.93	56.530	150.00	M P
16/12/2003	232	4	28.336	13.240	57.457	1.53	1.88	3.51	6.95	15.10	18.47	13.281	1.82	57.145	150.10	M P
Total (N)			10.000	10.000	10.000	10.00	10.00	10.00	10.00	10.000	10.000	10.000	10.00	10.000		
Mean			28.161	13.556	57.864	1.55	1.92	3.68	7.35	14.605	17.288	13.622	1.84	57.457		
Range			3.214	.489	2.061	.13	.12	.31	.85	3.133	2.988	.505	.14	1.888		
Sigma			.912	.169	.568	.04	.04	.11	.32	.900	1.027	.179	.04	.511		
LSD (5%)			2.582	.480	1.609	.12	.11	.31	.91	2.548	2.907	.507	.11	1.446		
%Coef.of.Variation			3.240	1.250	.982	2.64	2.05	2.95	4.36	6.165	5.942	1.314	2.14	.890		
CP			.587	.481	.604	.55	.50	.48	.44	.580	.485	.470	.57	.616		
CPk			.539	.339	.581	.36	.34	.41	.40	.488	.391	.307	.41	.604		

Legend: TP-Thermoplasticity CR-Cure Rate TR-Trends R-Reverting,P-Plateau,M-Marching R-Results P-Pass,F-Fail,?-Not Known
 CP-Process Capability CPk-Process Capability Index



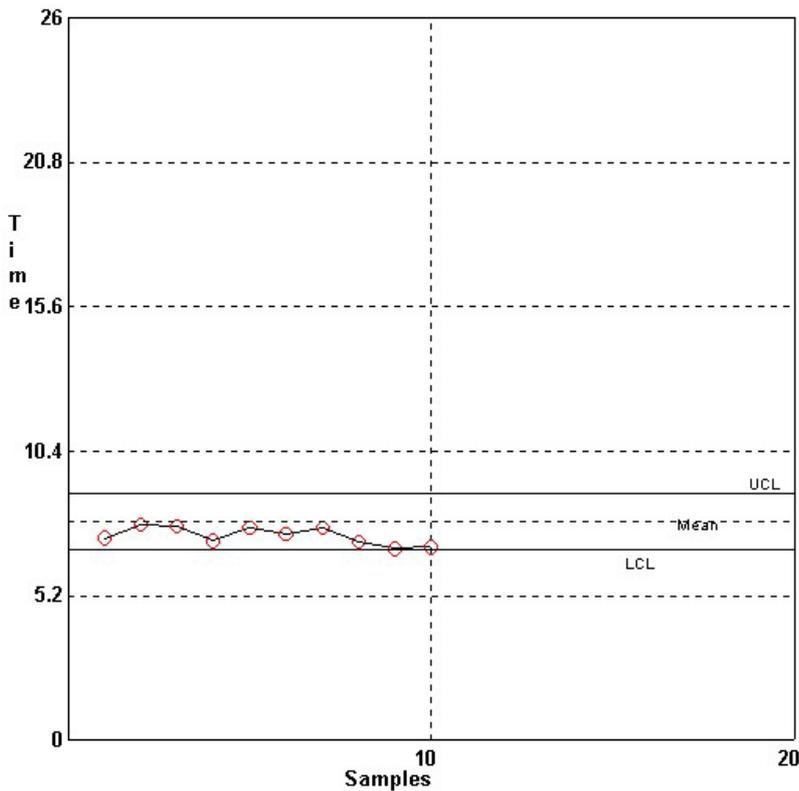
tc90 Time

RHEO-SOFT From - 16/12/2003 To - 16/12/2003 DETAILED 31/12/2003 Page No. 1
 Stock : TEST ARC± : 3 degree Temperature : 150.00 °C
 Run Duration : 12 Min. (X-axis-Time) Torque Range : 100 lb in

Lower Range 6.85
 Higher Range 8.85

Date	Specimen Srl	Batch	tc90 Time
16/12/2003	219	1	7.27
16/12/2003	221	2	7.74
16/12/2003	222	3	7.69
16/12/2003	223	4	7.17
16/12/2003	224	5	7.65
16/12/2003	225	6	7.41
16/12/2003	227	8	7.65
16/12/2003	229	1	7.12
16/12/2003	230	2	6.89
16/12/2003	232	4	6.95

Total (N) 10.000
 Mean 7.35
 Range .85
 Sigma .32
 LSD (5%) .91
 %Coef.of.Variation 4.36
 CP .44
 CPk .40



STATISTICAL QUALITY CONTROL (SQC)

RHEO-SOFT		From - 16/12/2003		To - 16/12/2003		31/12/2003		Page No. 1	
Stock : TEST		ARC± : 3 degree				Temperature : 150.00 °C			
Run Duration :12 Min. (X-axis-Time)						Torque Range : 100 lb in			
	Minimum Torque	Maximum Torque	ts2	ts5	tc50	tc90	QC1 Torque	QC2 Time	QC3 Torque
Total (N)	10.000	10.000	10.00	10.00	10.00	10.00	10.000	10.00	10.000
Upper Reading	13.729	58.854	1.64	2.00	3.82	7.74	13.787	1.93	58.418
Lower Reading	13.240	56.793	1.51	1.88	3.51	6.88	13.281	1.80	56.530
Mean	13.556	57.864	1.55	1.92	3.68	7.35	13.622	1.84	57.457
Range	.489	2.061	.13	.12	.31	.85	.505	.14	1.888
Sigma	.169	.568	.04	.04	.11	.32	.179	.04	.511
LSD (5%)	.480	1.609	.12	.11	.31	.91	.507	.11	1.446
%Coef.of.Variation	1.250	.982	2.64	2.05	2.95	4.36	1.314	2.14	.890
1 Sigma % Reading	70.000	80.000	80.00	70.00	70.00	60.00	80.000	70.00	80.000
2 Sigma % Reading	100.000	100.000	90.00	100.00	100.00	100.00	100.000	90.00	100.000
3 Sigma % Reading	100.000	100.000	100.00	100.00	100.00	100.00	100.000	100.00	100.000
CP	.481	.604	.55	.50	.48	.44	.470	.57	.616
CPk	.339	.581	.36	.34	.41	.40	.307	.41	.604

