NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

NATO STANDARDIZATION AGENCY (NSA)
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1110 BRUSSELS

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See CNAD AC/310 STANAG distribution

STANAG 4540 PCS (EDITION 1) – EXPLOSIVES, PROCEDURES FOR DYNAMIC MECHANICAL ANALYSIS (DMA) AND DETERMINATION OF GLASS TRANSITION TEMPERATURE

Reference: AC/310-D/191, dated 24 September 2001

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page (iii) is promulgated herewith.

2. The reference listed above is to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page (iii) of the STANAG and, if they have not already done so, advise the Defence Support Division through their national delegation as appropriate of their intention regarding its ratification and implementation.

Jan H ERIKSEN
Rear Admiral, NONA
Director, NSA

Enclosure:
STANAG 4540 (Edition 1)

STANAG 4540
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

NATO STANDARDIZATION AGENCY
(NSA)

STANDARDIZATION AGREEMENT
(STANAG)

SUBJECT: EXPLOSIVES, PROCEDURES FOR DYNAMIC MECHANICAL ANALYSIS (DMA) AND DETERMINATION OF GLASS TRANSITION TEMPERATURE

Promulgated on 23 August 2002

Jan H ERIKSEN
Rear Admiral, NONA
Director, NSA

STANAG 4540
(Edition 1)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Director, NSA under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "In NATO Standardization, the fulfilment by which a member nation formally accepts, with or without reservation, the content of a Standardization Agreement" (AAP-6).

5. Implementation is "In NATO Standardization, the fulfilment by a member nation of its obligations as specified in a Standardization Agreement" (AAP-6).

6. Reservation is "In NATO Standardization, the stated qualification by a member nation that describes the part of a Standardization Agreement that it will not implement or will implement only with limitations" (AAP-6).

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page (iii) gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page (iv) (and subsequent) gives details of reservations and proprietary rights that have been stated.

FEEDBACK

8. Any comments concerning this publication should be directed to NATO/NSA - Bvd Leopold III, 1110 Brussels - BE.

STANAG 4540
(Edition 1)

NAVY/ARMY/AIR

NATO STANDARDISATION AGREEMENT
(STANAG)

EXPLOSIVES, PROCEDURES FOR DYNAMIC MECHANICAL ANALYSIS (DMA) AND DETERMINATION OF GLASS TRANSITION TEMPERATURE

Annexes:

A. Definition of terms used
B. Test Procedures
C. Sample Preparation
D. Data Exchange Format

Related Documents: None

AIM

1. The aim of this agreement is to standardize test procedures for Dynamic Mechanical Analysis of explosives to provide acceptable data for international exchange between NATO nations.

AGREEMENT

2. Participating nations agree that the procedures in Annex B shall form the basis on which dynamic mechanical analysis of explosives shall be undertaken.

DEFINITIONS

3. Definitions of the terms used in the test procedures are given at Annex A.

SCOPE OF THE AGREEMENT

4. a. This document defines a standard test technique for the dynamic mechanical analysis (DMA) of solid propellant and other explosive materials undergoing deformation. To characterize a material, tests should be conducted at several temperatures.

b. A dynamic test is one in which the material is subjected to a cyclic, usually sinusoidal, deformation with the stress and strain being recorded continuously. The stress and strain information is analyzed to produce the two moduli, G′ and G′′ (or E′ and E′′). It is normal to measure these parameters as a function of temperature and often as a function of frequency of deformation.

c. The STANAG defines requirements, recommendations and/or factors to consider for each section of the test procedure. Every detail of the test that deviates from the requirements shall be reported. Recommendations and factors to consider are only provided for information purposes.

d. Within this procedure reference is made to “Glass Transition Temperature” of an explosive material. This method is the preferred method for the determination of this temperature.

e. The standard units for measurement are SI units.

WARNING

5. This STANAG calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken.

IMPLEMENTATION OF THE AGREEMENT

6. This STANAG is considered to be implemented by a nation when nations has issued the necessary orders/ instructions to adopt the text procedures here presented.

DEFINITION OF TERMS USED

Strain (ε)

1. The change in a dimension divided by the original value of the same dimension. This definition is sometimes referred to as engineering strain. Strain is normally expressed as a percentage. In DMA testing very small strains are generated by longitudinal vibration, twisting or bending of the test piece.

Stress (σ)

2. The applied load (F) divided by the original corresponding cross sectional area (A0). This is sometimes referred to as nominal stress, engineering stress or uncorrected stress.
Units are MPa.

Modulus (G, E)

3. The modulus is the ratio of the resultant stress to the applied strain. This quantity is represented by a complex number.

Storage Modulus (G′, E′)

4. The storage modulus is the component of the modulus in phase with the applied strain, it reflects the elastic energy stored in the material.

Loss Modulus (G″, E″)

5. The loss modulus is the component of the modulus which is ninety degrees out of phase with the applied strain, it reflects the energy dissipated during deformation.

Delta (δ)

6. Delta is the phase angle between the applied strain and the resultant stress.

Tan Delta (tan δ)

7. Tan δ is the ratio of the loss modulus to the storage modulus. This is also called the loss tangent.

Glass Transition Temperature (Tg)

8. The Glass Transition Temperature is the temperature at which main chain molecular motion ceases.

NOTE: If other data are used, definitions and data reduction procedures should be provided.

TEST PROCEDURES

1. TEST APPARATUS

a. Specimen measurement equipment

(1) Requirements: Accuracy: The measurement equipment shall measure to a minimum linear accuracy of 0.5% of the smallest dimension.

(2) Recommendation: Equipment that imposes no distortion in the process of measuring the specimen should be sought. This is critical to accuracy of the calculations.

b. Environmental conditioning

(1) Pre-conditioning requirements:

Temperature: Capable of maintaining selected temperature level to ± 2°C
Relative Humidity: Capable of maintaining the (if controlled) selected level to ± 5%. Selected levels may be between 10% and 90% RH.

(2) Test chamber requirements:

Temperature: Capable of maintaining selected temperature level to ± 1°C
Relative Humidity: Capable of maintaining the (if controlled) selected level to ± 5%. Selected levels may be between 10% to 90% RH.

c. Test Machine

(1) The dynamic mechanical analysis of materials can be conducted in a number of ways. The basic principle is to apply an oscillatory motion and measure the material response. The mode of testing is dependent on the type of machine used. Three modes of deformation are in common use: tension, torsion and flexure (pull, twist and bend). For the tension test a uniaxial test machine is fitted with an actuator to apply small displacements to the sample at a range of frequencies. For the torsional shear test a rectangular bar or cylindrical rod is gripped at one end. A sinusoidal deformation is applied while the resultant torque is measured at the other end of the sample. The third mode of test uses a three point bend/flexure geometry, the sample is held at each end and a sinusoidal deformation is applied to the centre. Some machines have multiple deformation modes.

(2) In all three modes the applied deformation (strain) and the resultant load measurements are used to calculate the two components of the modulus and the phase angle. Modes with homogeneous strain fields provide results requiring fewer assumptions (see below 4. Data Reduction).

(3) Accuracy: As there is a wide variation in sample sizes used with the various machines it is not possible to give figures for the accuracy of the results.

(4) Factors to consider: During temperature sweeps of polymeric materials there will be a considerable change in the length of the sample. It is essential that this change can be accommodated otherwise the sample will buckle and affect the results. A machine that cannot accommodate these changes is not recommended. (Some machines measure this change in length to allow the determination of the coefficient of thermal expansion.)

d. Specimen holding grips

(1) Requirement: For the small samples generally used in these tests it is important that the grips securely hold the sample.

(2) Factors to consider: Any slackness in the gripping system will result in an underestimate of the elastic modulus. When the grips are tightened there is a tendency for the sample to buckle as it is squeezed from the grips, compensation must be allowed for this bending. If a temperature sweep is to be conducted then the sample grips should be tightened at the lowest temperature used. Tightening should be done after the sample and gripping system have come to equilibrium at that temperature.

e. Test data digital equipment

Factors to consider: Rate: Increases in the sampling rate provide finer resolution for automatic data reduction.

2. TEST SPECIMEN

a. The method of preparation of the test specimen from bulk quantities may vary depending on the material, availability of equipment and existing procedures. Guidance on sample preparation is given in ANNEX C.

b. Specimen configuration

The sample geometry used in the test will be in the form of a rod or a rectangular bar. Size and shape will be dependant on the machine being used. The test specimen will be either cast, extruded exactly to size or produced in a large block which is subsequently machined to size.

A typical sample configuration would be a rectangular bar of material 10mm x 6mm x 50mm. The configuration must be reported.

c. Environmental conditions

Test specimens shall be carefully packed flat on plane surfaces to prevent distortion and damage. The samples shall be stored at a temperature and relative humidity that will not significantly change the original mechanical properties. Because of the small size of the sample extreme care must be taken to minimise exposure, which would alter the mechanical properties of the specimen.

Preferably, specimen preparation shall be carried out in an air-conditioned and humidity-controlled environment. Unless they are to be tested within two hours, the specimens should be closely wrapped or similarly protected to prevent moisture contamination. Inclusion of a humidity indicator on the bulk sample is recommended.

3. TEST METHOD

a. Several machines are available to conduct DMA testing. It is usually necessary to adopt one manufacturer's testing instructions as a local standard.

A specimen will be mounted in the testing frame and tested according to the machine manufacturer's instructions. For a typical shear test the materials will be tested within the temperature range -120°C to 100°C at three fixed frequencies (0.1, 1.0 and 10 radians per second). A sufficiently small strain should be applied to the material such that it remains in the linear portion of the stress/strain curve. This will typically be about 0.1% strain in the shear mode. The storage and loss moduli, G' and G'', and the loss tangent, $\tan \delta$, should be

recorded at suitable temperature increments throughout the test (5°C or less). The sample should be held at each temperature for at least three minutes. If unclear about the thermal equilibrium conditions an isothermal test should be conducted to determine the soak time.

b. Conditioning procedure

Samples shall be conditioned at the test temperature (or the lowest temperature of a sweep) for fifteen minutes prior to commencement of the test.

Recommendation: The temperature control of the machine test chamber should be accurate to within ± 1°C.

c. Equipment calibration procedure

Calibration procedures are different for the various machines. The machine manufacturer's calibration instruction should be performed at regular intervals; both stress and strain errors must be within the manufacturer's specification for the machine. It is normal to check the load system with a fixed standard weight every month.

Recommendation: The temperature measurement system should be calibrated regularly.

d. Specimen dimensional measurement device

Requirement: Accuracy to be ensured by calibration against gauge blocks and checked at least every three months.

e. Testing

Manufacturer's procedures for testing should be followed. Any deviation from these procedures should be noted.

Recommendation: Where a thermal ramp is used consideration must be given to the temperature lag in the sample.

f. Specimen examination

All samples should be examined for visible flaws before and after testing. If samples exhibit flaws, said information should be documented. Flaws include voids, low-density areas, binder-rich areas, oversized particles, foreign matter, agglomerates etc.

g. Specimen installation and positioning

The sample shall be placed in the grips with a minimum amount of handling. It must be mounted and aligned as instructed by the machine manufacturer.

h. Data recording

The data exchange format (ANNEX E) should be filled in as appropriate.

4. DATA REDUCTION

a. Dynamic mechanical analysis is the preferred method for determination of the glass transition temperature of explosive materials. This is due to the fact that Tg represents the temperature at which a mechanical transition occurs and therefore should be measured by a mechanical method.

The measurement of Tg is obtained from the peak in the loss modulus. The value of the Tg is frequency and amplitude dependant and if possible the relation between such conditions must be stated.

An example curve for a typical double base propellant is shown in Figure 1. In this figure G′, G″ and tan δ are shown plotted against temperature.

b. Data reporting

Data shall be generated to complete the data exchange format at a frequency of about 1 Hz.

5. CAUTIONS

a. The sample will expand with temperature during testing and if this is not accommodated spurious results will be obtained.

b. Results are influenced by the mode of deformation employed and the clamping system used by the test machines.

6. REFERENCE

T Murayama, Dynamic Mechanical Analysis of Polymeric Materials, Elsevier Applied Science Publishers 1978,

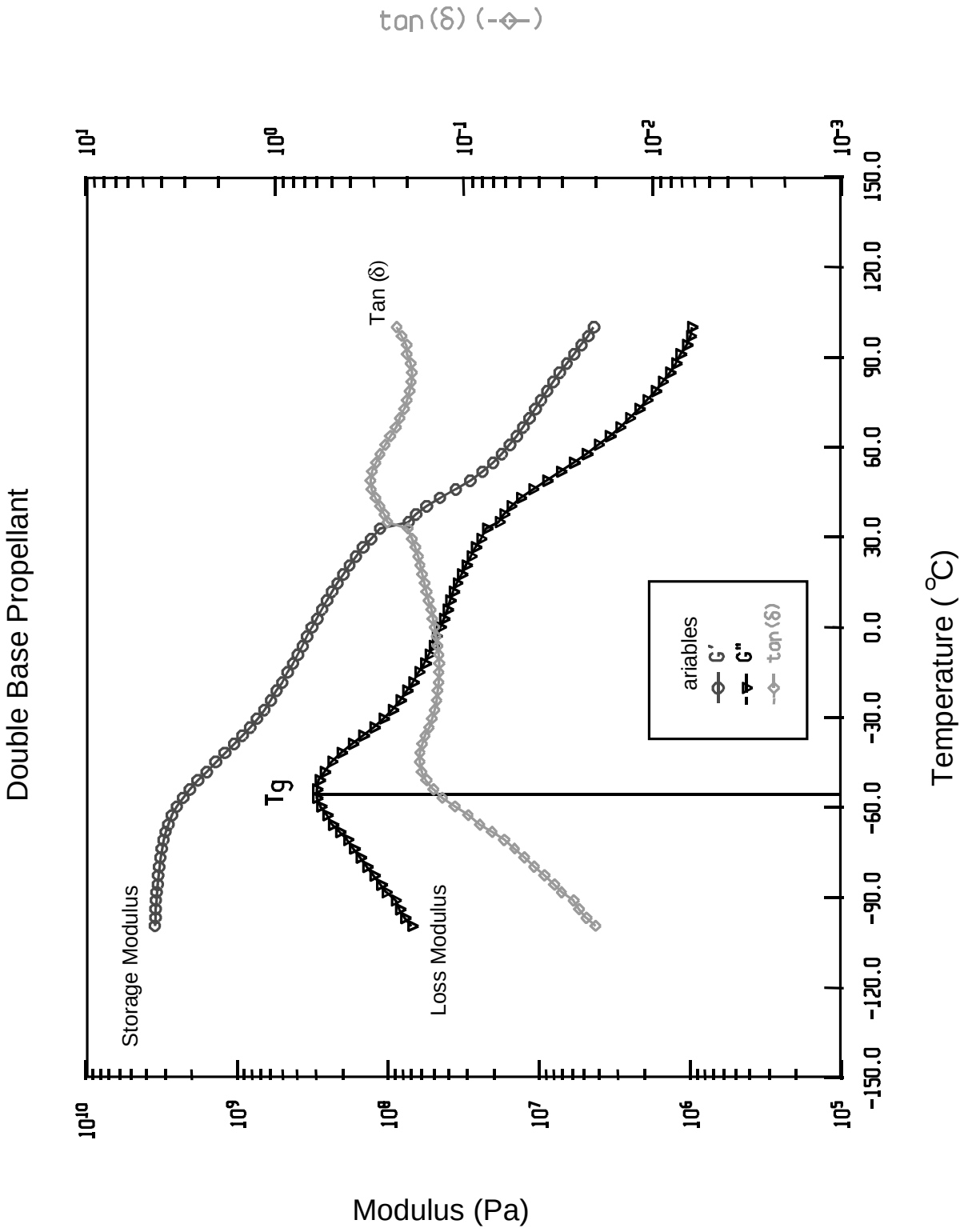

Figure 1

SAMPLE PREPARATION

1. PREPARATION OF BULK EXPLOSIVE

Bulk quantities of explosives from charge sections, large blocks or cartons (smaller block samples retained from manufacture) may need to be reduced to a size suitable for laboratory use by one of the following methods: sawing, machining, slicing, guillotining and/or wire cutting.

To avoid binder diffusion affected or otherwise damaged surfaces, cutting shall be conducted in such a manner that the "skin" on the surface of a block of explosive material shall be removed.

a. Recommendations:

A minimum depth of 12.5mm should be removed from all surfaces of the block or bulk sample. However in many cases this may not be possible.

Temperature: As required for machinability and safety considerations however temperatures other than ambient should be reported.

Relative Humidity: < 50% (<20% for hygroscopic materials)

Tool Speed: As a function of material modulus

b. Factors to consider: Storage conditions and orientation of specimens shall be carefully maintained. Inadequate control of the storage conditions can lead to wide variation in test results. In particular many explosive materials are sensitive to variations in temperature and humidity.

NOTE: The sample sizes used during testing will be dependant on the particular test machine used. The following are general guidelines which should be appropriate to all test specimens.

2. METHOD 1: MACHINING

a. Requirements

The specimen shall conform in geometric shape and dimensions to those specified by the instrument manufacturer. A typical sample size would be 6mm x 10mm x 50mm.

Temperature: As required for machinability and safety considerations. Temperatures other than ambient should be reported.

Tool Speed: As a function of material modulus.

Dimensional Tolerances: To a minimum accuracy of 0.5% of the smallest dimension.

b. Factors to consider

Due to changes in the material, feed and cutting rates may have to be adjusted to maintain required average dimensions. If the specimen preparation area has a relative humidity greater than 50%, exposure of the specimens to the environment shall be minimized.

3. METHOD 2: DIE CUTTING, SLICING AND GUILLOTINING

a. Requirements

The specimen shall conform in geometric shape and dimensions to those specified by the instrument manufacturer. A typical sample size would be 6mm x 10mm x 50mm.

Temperature: As required for machinability and safety considerations. Temperatures other than ambient should be reported.

Tool Speed: As a function of material modulus.

Dimensional Tolerances: To a minimum accuracy of 0.5% of the smallest dimension.

b. Factors to consider

If the specimen preparation area has a relative humidity greater than 50%, exposure of the specimens to the environment shall be minimized.

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NATO AOP-7 DATA EXCHANGE FORMAT (Side A)

Dynamic Mechanical Analysis

Report Reference Number:
(Unique Reference Number)

Page __ of __Page(s)

TEST CONDITIONS

Initial/Final Temperature (^{o}C):
(Temperature range)

Conditioning Temperature (^{o}C):

Conditioning Time (min):

Osc Amp (^{o}, mm or %):
(Amplitude of oscillation)

Frequency (Hz; rad/s):
(Frequency of oscillation)

Temperature Rate (^{o}C min^{-1}):
(Rate of temperature sweep)

Machine Type:
(Name and model number of machine)

Grip Type:
(Name of grip)

Test Type:
(Type of DMA test)

SPECIMEN INFORMATION

Dimensions (mm): (Mean specimen length, width & thickness)
Length:
Width:
Thickness (diameter):

Length Correction:
(Effective length extension of specimen)

Form:
(Specimen geometry descriptor)

Identification:
(Trade name and/or Identity code)

Preparation Method:
(Specimen preparation procedure)

Manufacturer Method:
(Material processing technique)

Source:
(Name of manufacturer)

Lot, Batch or Consignment Number:
(Same as on material description sheet)*

Conditioning history:
(Period specimen was conditioned)

TEST SITE INFORMATION

Laboratory:
(Name of Laboratory)

Date:
(Date that form was completed)

Date Tested:
(Date of test period)

Test Procedure:
(Name of test procedure used)

AOP-7 Test Procedure Number :
(Test procedure number)

POC:
(Point of contact)

COMPOSITION

(Component and percentages)

* If the specimens have been conditioned in any way that makes them different from those usually indicated by this Lot Identification Number, add a suffix to the Lot or ID Number to indicate this difference, e.g if Lot RAD890522 was aged for 30 days at 60ºC it could be written as: RAD890522-30D60C.

NATO AOP-7 EXCHANGE FORMAT (Side B)

Dynamic Mechanical Analysis

ANNEX D to
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(Edition 1)

Report Reference Number:
(Unique Reference Number)

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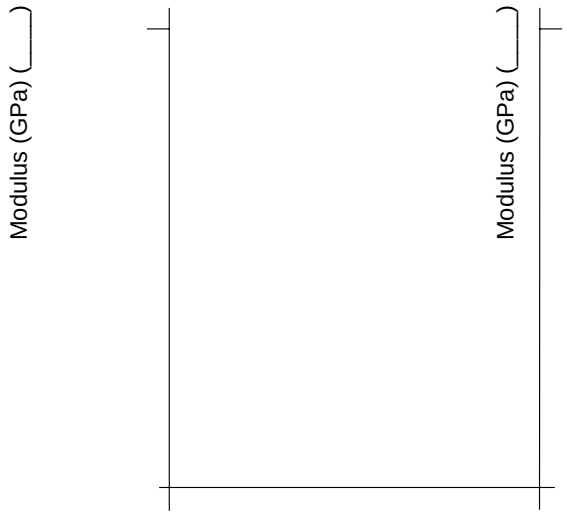
RESULTS

T (^{o}C)	E' (GPa)	E" (GPa)	G' (GPa)	G" (GPa)	Tan δ	f (Hz; rad/s)
(Specimen temperature)	(Storage Tensile Modulus)	(Loss Tensile Modulus)	(Storage Shear Modulus)	(Loss Shear Modulus)	(E"/E' or G"/G')	(Oscillation frequency)

DMA SPECTRUM

(Scale all axes)



Tg = at Hz
(Glass transition temperature)

Comments:

Data Sent To: (Name and address of person receiving this information)