



Standard Test Method for Tension Testing of Calcium Phosphate and Metallic Coatings¹

This standard is issued under the fixed designation F 1147; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers tension testing of calcium phosphate and metallic porous coatings adhering to dense metal substrates at ambient temperatures. It assesses the degree of adhesion of coatings to substrates, or the internal cohesion of a coating in tension normal to the surface plane.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 633 Test Method for Adhesion or Cohesive Strength of Flame-Sprayed Coatings²

E 4 Practices for Force Verification of Testing Machines³

E 6 Terminology Relating to Methods of Mechanical Testing³

E 8 Test Methods for Tension Testing of Metallic Materials³

3. Terminology

3.1 The definitions of terms relating to tension testing appearing in Terminology E 6 shall be considered as applying to the terms used in this test method.

4. Summary of Test Method

4.1 The tensile test method consists of subjecting a specimen assembly composed of one coated and one uncoated component to a tensile load. In the case of the calcium phosphate coatings, the components to be tested must be bonded together by use of a polymeric adhesive. In the case of the metallic coatings, the components may either be bonded

with the adhesive, or sintered together. The adhesive may be in film form or bulk form, but it must have a minimum bulk tensile strength of 34.5 MPa (5000 psi).

4.2 The tensile load must be applied normal to the plane of the coating utilizing a tension machine which is capable of determining the maximum strength of the coating or coating attachment to the substrate interface.

5. Significance and Use

5.1 The tensile test method is recommended for tension testing of calcium phosphate/substrate or porous metal coating/substrate combinations and can provide information on the adhesive or cohesive strength of coatings under (uniaxial) tensile stress.

5.2 The test method may be useful for comparative evaluation of adhesive or cohesive strengths of a variety of types of coatings. Coatings may be applied using a variety of methods, including but not limited to plasma-spraying and sintering. Information developed using this method may be useful for certain quality control and design purposes.

5.3 The test should not be considered to provide an intrinsic value for utilization directly in making calculations such as determining the ability of a coating to withstand specified environmental stresses.

5.4 Processing variables such as substrate preparation prior to coating, surface texture, coating technique variables or postcoating heat treatment variables may introduce a significant effect on the results of the tension test. The specimen being evaluated must be representative of the actual end use coating.

6. Apparatus

6.1 *Testing Machines*—Machines used for testing shall conform to the requirements of Practices E 4. The loads used in determining tensile strength and yield strength shall be within the loading range of the testing machine as defined in Practices E 4.

6.2 Gripping Devices:

6.2.1 *General*—Various types of grips may be used to transmit the load applied to the specimens by the testing machine. To ensure axial tensile stress, it is important that the specimen axis coincide with the centerline of the heads of the testing machine and that the coating test plane be perpendicular

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² *Annual Book of ASTM Standards*, Vol 02.05.

³ *Annual Book of ASTM Standards*, Vol 03.01.

to the axial load. Any departure from this requirement (that is, any eccentric loading) will introduce bending stresses that are not included in the usual stress calculation (force/cross-sectional area).

6.2.2 *Yoke and Dowel Pin Grips*—A schematic diagram of a typical gripping device for specimens with holes drilled for pin loading is illustrated in Fig. 1. There should be two perpendicular pins in order to maximize off axis loading.

7. Materials

7.1 *Adhesive Bonding Agent*—A polymeric adhesive bonding agent in film form, or filled viscous adhesive cement, when used, shall be identified and shall meet the following requirements.

7.1.1 The bonding agent shall be capable of bonding the coating on the test specimen components with a tensile strength that is at least 34.5 MPa (5000 psi) or as great as the minimum required adhesion or cohesion strength of the coating.

7.1.2 In instances where porosity extends to the coating substrate interface, the bonding agent shall be sufficiently viscous and application to the coating sufficiently careful to assure that it will not penetrate through the coating to the substrate. The FM 1000 Adhesive Film⁴ with a thickness of 0.25 mm (0.01 inc.) has proven satisfactory for this test. If a

material other than FM 1000 is used, or the condition of the FM 1000 is unknown, it must be tested to establish its equivalence fresh FM 1000. Testing should be performed without the presence of the coating to establish the performance of the adhesive.

8. Test Specimens

8.1 *General:*

8.1.1 In order to ensure precision and accuracy in test results, it is important that care be exercised in the preparation of specimens, both in machining and in the case of multi-part specimens, in the assembly. Specimen components must be properly aligned in order that generated stresses be purely axial, that is, normal to the coated surface.

8.1.2 *Tensile-Type Specimens*—Three general types of test specimens are illustrated in Figs. 2 and 3. A complete, assembled test assembly consists either of two solid pieces; one with a coated surface and the other with an uncoated surface or three solid pieces; two with uncoated surfaces and one with a coating applied on one side. The uncoated surface may be roughened to aid in the bonding of the adhesive.

⁴ Available from Cytec, Harve Grace, Maryland.

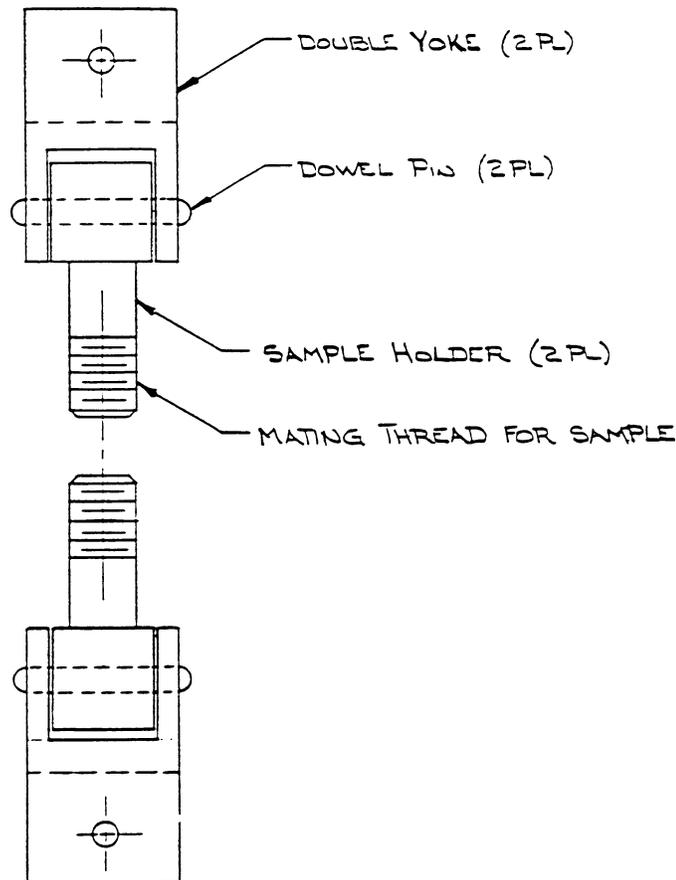


FIG. 1 Yoke and Dowel Pin Grips for Tensile Testing Porous Surfaces

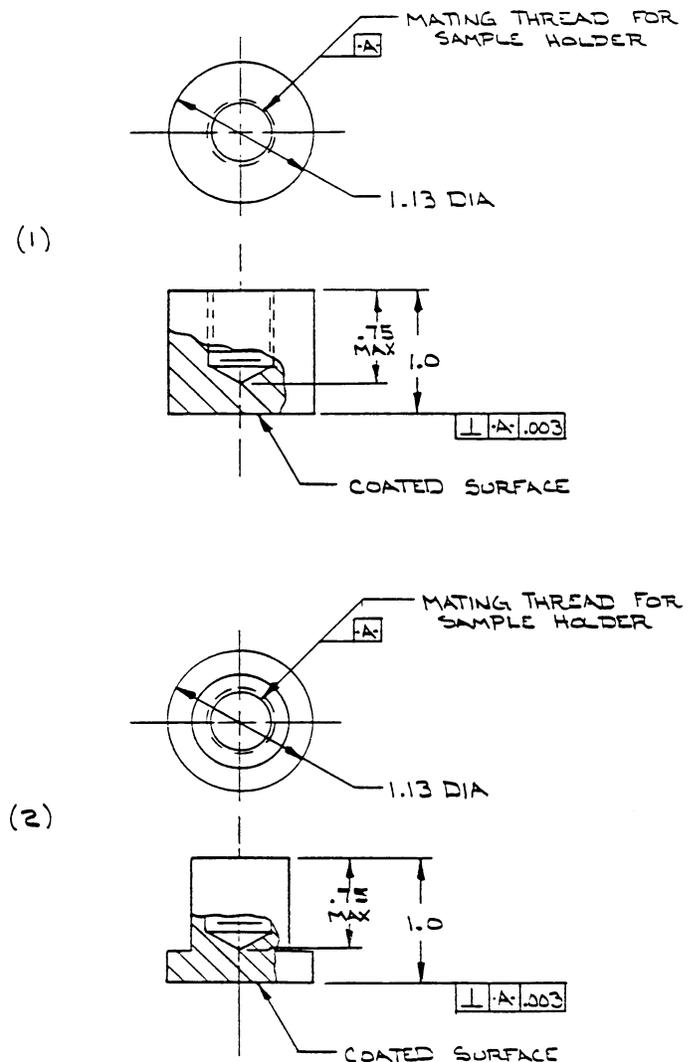


FIG. 2 Tensile Specimens for Testing Porous Surfaces

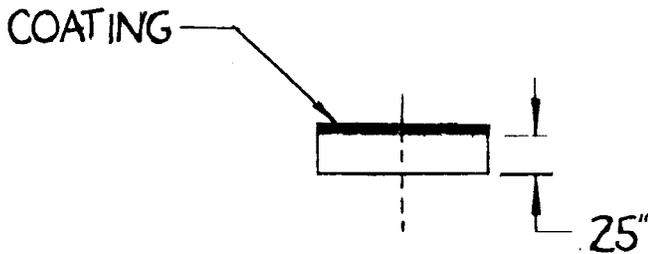
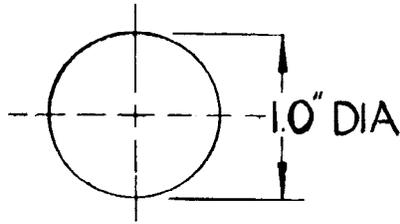


FIG. 3 Smooth Tensile Specimens

8.1.3 The cross-sectional area of the substrate upon which the coating is applied shall be nominal 5.07 cm² (0.78 in²). When specimens of another cross-sectional area are used, the data must be demonstrated to be equivalent to a 5.07 cm² standard cross-sectional area, and the specimen size should be reported.

8.1.4 All test specimens for coating characterization shall be prepared from indicative coating lots, using production feed-stock lots and be coated on the same equipment used for actual implants.

8.2 Specimen Coating Preparation:

8.2.1 Coatings may be applied by any one of a number of techniques. The coating should consist of a layer which is mechanically or chemically attached and covers the surface.

8.2.2 All thermal treatments normally performed on the devices should be performed on the test specimens.

8.2.3 If employed, passivation and sterilization techniques should be consistent with those used for actual devices.

8.2.3.1 If the passivation and sterilization processes can be shown not to influence the tensile strength, these steps may be eliminated.

8.2.4 Inspection - Before testing, visual inspections should be performed on 100 % of the test specimens. Lack of coating in highly stressed regions, as well as non-uniform coating appearance, shall be cause for specimen rejection.

9. Procedure

9.1 Specimens tested with FM1000 adhesive shall be prepared as follows:

Curing the adhesive - The test results achieved are greatly dependent upon the adhesive used and the way in which it is cured. One suggested adhesive commonly used with calcium

phosphate coatings is FM-1000 having a thickness of 0.25 mm.(0.01 in.). This material has successfully been cured using the following cycle: Where testing porous coatings, more than one layer of glue may need to be employed.

NOTE 1—In the case of porous coatings, care must be taken to guarantee that the adhesive does not bond to the substrate.

9.1.1 Align the adhesive with the surface of the coating, taking precautions to align the adhesive in the center of the coating.

9.1.2 Apply a constant force using a calibrated high temperature spring, resulting in a stress of 0.138 MPa.(20 Psi.) between the coating and the opposing device that will test the coating.

9.1.2.1 Care must be taken to maintain alignment of the coating and the matching counterface during the curing of the adhesive.

9.1.3 Place the assembly in an oven and heat at 176°C (350°F) for 2-3 h.

9.1.3.1 The exact amount of time necessary to cure the adhesive will need to be determined by each user, as oven temperature may vary with load size and oven type. It is suggested that the curing cycle be optimized without the coating present, first.

9.1.4 Remove the cured assembly from the oven and allow it to cool to room temperature.

9.1.5 Remove all excess glue which has protruded from the coated surface. This process must not compromise the integrity of the sample.

9.2 Place the specimen assembly in the grips so that the long axis of the specimen coincides with the direction of applied tensile load through the centerline of the grip assembly.

9.3 Apply a tensile load to each test specimen at a constant rate of cross-head speed of 0.25 cm./min.(0.10 in./min.). The test should be continued until complete separation of the components has been achieved. Record the maximum load applied.

10. Calculation

10.1 Calculate the substrate area upon which the coating is applied to the nearest 0.006 cm² (0.001 in.²). Record peak (failure) load and calculate failing stress in MPa (psi) of adhesive bond area as follows:

$$\text{Adhesion or cohesion strength} = \text{maximum load/cross-section area} \tag{1}$$

11. Report

11.1 The report shall include the following information:

11.1.1 Identification of the materials used in the specimen, including bonding agent if used.

11.1.2 Identification of methods used to apply the coating including coating method, heat-treatment, or other data if available, including date, cycle number, and time and temperature of the run.

11.1.3 Dimensional data including the bond cross-sectional area and the thickness of the porous or other coated layer.

11.1.4 Number of specimens tested.

11.1.5 Report all values for the failure load, including maximum, minimum, and mean.

11.1.6 The mode of failure (for example, cohesive versus adhesive) for each test specimen.

12. Precision and Bias

12.1 *Precision (beaded F75)*—The precision of this method was established by an interlaboratory comparison among six (6) laboratories. The specimens tested were sintered-bead coatings of F75 (Co-Cr-Mo) alloy on F75 substrates. The population mean tensile strength was 4325 lbf/in² (29.8 MPa) for all samples tested by all laboratories.

12.1.1 *Repeatability*—For replicate results obtained by the same operator on nominally-identical test materials, the repeatability standard deviation (S_r) was 613 lbf/in² (4.23 MPa). Any two such results (would be expected to differ by more than) 1715 lbf/in² (11.8 MPa) only 1 time in 20.

12.1.2 *Reproducibility*—For independent results obtained by different operators working in different laboratories on nominally-identical test materials, the reproducibility standard deviation (S_R) was 772 lbf/in² (5.32 MPa). Any two such results (would be expected to differ by more than) 2160 lbf/in² (14.8 MPa) only 1 time in 20.

NOTE 2—The precision study under Section 12.1 was conducted using coupons which had a one square inch area (1.13 in. diameter). The bead tensile strength was intentionally lowered for this round robin comparison to produce coating fractures rather than glue failures in the coupons.

12.2 *Precision (HA Coating)*—The precision of this method was established by an interlaboratory comparison among seven (7) laboratories. The substrate of the specimens tested was

Titanium -6 Aluminum -4 vanadium, which was hydroxylapatite coated. The mean tensile strength was 8405 lbf/in² (58.0) MPa for all samples tested by all laboratories.

12.2.1 *Repeatability*—For replicate results obtained by the same operator on nominally-identical test materials, the repeatability standard deviation S_r was 1016 lbf/in² (7.0 MPa). Any two such results (would be expected to differ by more than) 2845 lbf/in² (19.6 MPa) only one time in 20.

12.2.2 *Reproducibility*—For independent results obtained by different operators working in different laboratories on nominally-identical test materials, the reproducibility standard deviation (S_R) was 1758 lbf/in² (12.1 MPa). Any two such results (would be expected to differ by more than) 4922 lbf/in² (33.9 MPa) only one time in 20.

NOTE 3—The precision study under Section 12.2 was run using a cross-head speed of 0.05 in/min (0.12 cm/min).

12.3 *Bias*—Since the measurement of porous-coating tensile strength by this method is a destructive test measurement unique to each individual test sample, no independent determination of a “correct” or “reference” value is possible. Thus, no statements regarding the deviation of values, measured in accordance with these procedures, from accepted values is possible.

13. Keywords

13.1 ceramic materials; hydroxylapatite; tribasic calcium phosphate; tension testing; adherence; interface; porous coating

ANNEXES

(Mandatory Information)

A1. PROCEDURE FOR BONDING POROUS COATED TEST SPECIMENS USING 3M SCOTCH-WELD 2214-NMF STRUCTURAL ADHESIVE

A1.1 Surface to be bonded must be clean, degreased, and free of any loose particles/beads.

A1.2 A thin layer of the adhesive shall be applied evenly to the coated surface of the test coupons. A spatula or tongue depressor may be used as application device.

A1.3 Excess adhesive shall be squeezed out of the assembly using hand pressure while coated faces of the test coupons are buffed together.

A1.4 The specimen thus prepared will be placed in a suitable fixture. During curing, the fixture shall be able to maintain a mild pressure at the joint. At the same time, it shall also force bonding faces to remain parallel and maintain the axial alignment.

A1.5 The specimen joint shall be cured at $250 \pm 10^\circ\text{F}$ ($121 \pm 5.7^\circ\text{C}$) in air for 1.25 h (a sharp pointed pin must not be able to penetrate the adhesive joint after optimum curing).

A1.6 Air cool to room temperature prior to testing.

A2. PROCEDURE FOR BONDING POROUS COATED TEST SPECIMENS USING FM 1000 FILM ADHESIVE

A2.1 Clean the specimen holders. If the holders have test coupons on them from a previous test, place the holders in the oven at 270°C for 1 h.

A2.1.1 After 1 h, put oven gloves on and take the holders out of the oven.

A2.1.2 Break the coupons off of the holders with gloved hands or with a pair of pliers.

A2.1.3 Cool the holders in water or air cool.

A2.1.4 The adhesive will stay on the holder; this will have to be removed. The adhesive is removed by polishing it off on a fiber wheel. If necessary, face off the holders in a lathe, as the faces must be parallel. The holders are then grit blasted.

A2.1.5 Test coupons to be run should not have any proud beads, nor should the beads extend past the side of the specimen.

A2.1.6 The holders and test coupons are placed in a set of pliers and dipped in trichlorotrifluoroethane, or equivalent, which is to clean and degrease the parts.

A2.1.7 The holders and test coupons are placed on tissues to evaporate the trichlorotrifluoroethane.

NOTE A2.1—Handle all of the test material with gloved hands because the test can vary with the amount of oil absorbed from the skin.

NOTE A2.2—Follow all manufacturer's instructions concerning the storage procedure and shelf life of the adhesive.

NOTE A2.3—**Precaution:** Handle the trichlorotrifluoroethane with care and use under a vent-hood.

NOTE A2.4—**Precaution:** Avoid inhalation of fumes during heating cycles.

A2.2 The adhesive that is used is FM 1000 film, 0.06 or 0.05 lb/sq ft produced by the American Cyanamid Company. Cut disks out of the adhesive to match the shape and dimen-

sions of the test coupon.

A2.3 Typical assembly of components will include placing one adhesive disk between the back of the sample coupon and the specimen holder and two disks between the porous coated surface of the coupon and the other specimen holders. The amount of glue (the thickness of the sheets, as well as the number of sheets) is selected by a balance between having enough glue to fully bond the specimen holder with the test coupon, but not so much as to force the glue to the coupon's substrate which would render the test invalid. The fixturing of the assembly should maintain alignment during the glue curing cycle. A small compressive pressure placed on the components helps to prevent sliding and misalignment. This is best accomplished with a spring in the fixturing. An excessive pressure or amount of glue must not be used as the glue will be squeezed out and distort test values.

A2.4 Turn the oven on and preheat to 177°C (350°F). When the oven reaches the temperature, place the assembled curing fixtures in the oven on a level surface.

A2.5 Maintain this temperature for 2.5 h.

A2.6 Allow curing holders to air cool to room temperature before removing specimens.

A2.7 Samples cured at elevated temperatures should be allowed to remain at room temperature for 24 h before testing. This time period will allow for the adhesive to full cure, eliminating variations that may result if the adhesive has not reached an equilibrium temperature.

NOTE A2.5—A fan can be used to speed up the cooling process.

APPENDICES

(Nonmandatory Information)

X1. RATIONALE

X1.1 This standard is needed to aid in the development of a high quality material for use in load-bearing implant applications. The influence of coatings on the resulting tensile behavior of the system must be viewed as a combination of the surface roughening treatments required to apply the coating,

the thermal effects of the coating process, and any other secondary treatments employed. The purpose of this specification is to provide the following information: 1) the influence of the above processing steps, and 2) the integrity of the coating and the coating/substrate interface.

X2. PRECISION AND BIAS (EXAMPLE)
X2.1 Precision and Bias

X2.1.1 Review of the Round Robin - Seven laboratories were involved in round-robin testing. Each laboratory was provided with six (6) specimens coated with hydroxylapatite and an appropriate amount of FM 1000.

X2.1.2 Table X2.1 shows the raw data generated from each laboratory.

X2.1.3 *h* Graph:

X2.1.3.1 The *h* value evaluates the consistency of the test results from laboratory to laboratory.

X2.1.3.2 There are three patterns in these plots. In one pattern, all values are either positive or negative. In the second pattern, there are roughly the same number of laboratories which exhibit positive values as those which exhibit negative values. In the third type, one laboratory exhibits a value which is opposite of the other laboratories. The first two types are considered normal.

X2.1.3.3 Fig. X2.1 shows the *h* values for each laboratory.

X2.1.4 *k* Graph:

X2.1.4.1 The *k* value evaluates the consistency of the test results within each laboratory.

X2.1.4.2 The pattern to look for in the *k* graph is one laboratory having a very large or a very small *k* value. High *k* values indicate imprecision. Very small *k* values indicate a very insensitive measurement scale or other measurement problems. A *k* value greater than 1 indicates greater variability than other laboratories.

X2.1.4.3 Fig. X2.2 shows the *k* values for each laboratory.

X2.1.5 *Precision Statistics*—The precision statistics are shown in Table X2.2. These statistics are also reported in Section 12.2.

X2.1.6 ASTM would like to acknowledge the following companies for their help in the round-robin testing: Bio-Coat, Bio-vac, EML, FDA, Howmedica, Osteonics, and Steri-OSS.

TABLE X2.1 ASTM F 1147 Round Robin, Tensile Strength (psi)^A

Company #1	Company #2	Company #3	Company #4	Company #5	Company #6	Company #7
9790	8945	10770	8612	5206 ^B	9786	7222
7430	8150	10986	7949	9495	9469	4897
5510	6680	11712	6841	8832	11383	8402
8390	7144	11330	7261	8965	9048	8459
6750	5230	10922	7911	8743	9208	5919
7330	4815	10680	8230	8515	10614	7735
7533 ± 1455	6502 ± 1241	11067 ± 387	7801 ± 646	8910 ± 366	9918 ± 907	7106 ± 1429

^AData previously published in ASTM F 1501.

^BValue excluded due to improper alignment.

h Values (Critical = 2.05)

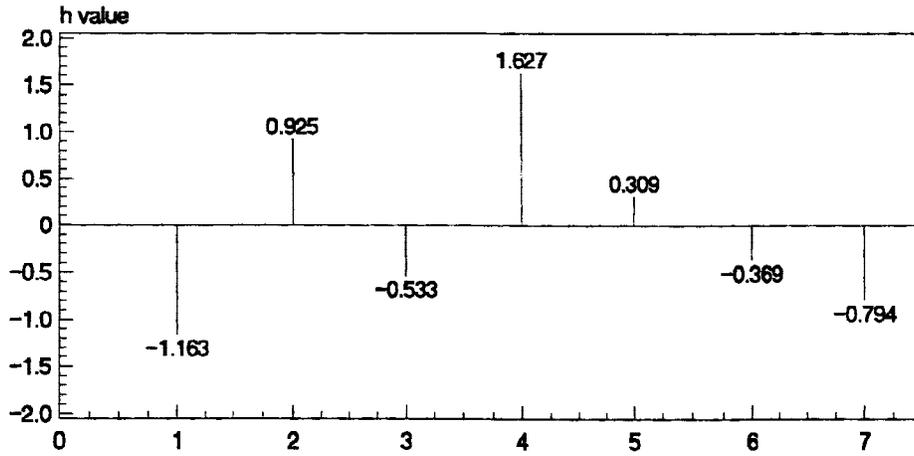


FIG. X2.1 h Values (Critical = 2.05)

k Values (Critical = 1.7)

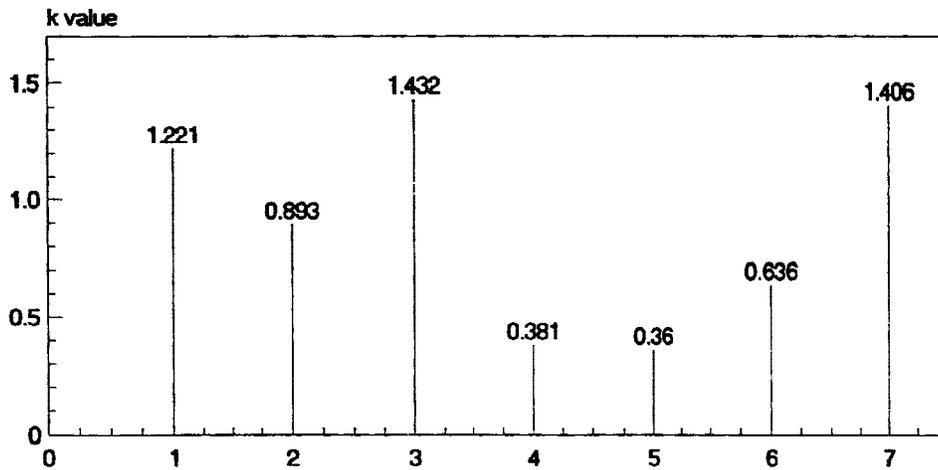


FIG. X2.2 k Values (Critical = 1.7)

TABLE X2.2 PRECISION STATISTICS

X	S _x	S _r	S _R	r	R
8405	1636	1016	1758	2845	4922

X = average of the cell averages
 S_x = standard deviation of the cell averages
 S_r = repeatability standard deviation
 S_R = reproducibility standard deviation
 r = 95 % repeatability limit, and
 R = 95 % reproducibility limit

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