INTRODUCTION

CRUDE OILS, petroleum products and lubricants are highly complex materials and enormous efforts have been spent by the oil companies throughout the world to characterize their chemical and physical properties with a high degree of precision and accuracy. The explosive growth in the availability of modern analytical instrumentation in the last three decades has significantly helped in the task of petroleum products analyses. These modern techniques have largely supplanted the classical "wet chemistry" types of analyses, which were used in the first half of this century [*1*]. However, there are still a few areas where some specific analyses need these older techniques.

ASTM Committee DO2 Petroleum Products and Lubricants has over most of this century led these efforts to develop more reliable and standard test methods to the point that in all corners of the world ASTM DO2 standards are considered as the final arbitrators of the quality of a petroleum related product. Other national and international standardization bodies such as IP in U.K., AFNOR in France, DIN in Germany, JIS in Japan, and ISO have also contributed significantly in developing standard test methods for the analyses of petroleum products. However, many of the latter standards are based on the ASTM DO2 standards.

There are about 350 ASTM test method standards available that involve a variety of analytical techniques to identify or quantify, or both, over 170 chemical and physical properties of crude oils, gasoline, reformulated gasoline, lubricating oils, additives, transmission fluids, lubricating greases, gear oils, aviation fuels, diesel and heating fuels, petroleum waxes, marine fuels, and other specialty petroleum products. These test methods are annually published in the *Annual Book of ASTM Standards* [*2*]. Additional methods continue to be developed in many oil company laboratories either to improve on the existing methods or to enable determination of other properties. Many of such methods remain proprietary to the oil companies.

An earlier manual on the significance of tests for petroleum products emphasized the rationale of specific tests conducted on different products [*3*]. However, this valuable manual did not include the details of any tests, but rather discussed the compositions of products and the primary quality characteristics that defined that material's use in applications.

It is certainly not the intention of this author to replace *Annual Book of ASTM Standards* or the other valuable manual with the current book, but rather to view it as a complementary material for the customer. The purpose of this book is to make available in one handy volume, the essential elements of all analytical tests used to characterize the petroleum products. It is of course critical for the testing laboratory personnel to be fully familiar with all the details of the tests they are performing. But it is also important for non-laboratory personnel to know at least the significance, advantages and limitations of particular tests used to characterize the product quality. Both the suppliers and the customers need to agree on the appropriate product quality specifications, and this can be done only by understanding the pros and cons of these tests. Product specifications not based on realistic testing capabilities can only lead to quality complaints and unhappiness on the part of both suppliers and customers. As such, we expect that this book will prove useful not only to the laboratory personnel, but also to the product specification writers, formulators, process engineers, researchers, and marketing staff in understanding the importance of these tests as well as their limitations, so that sound conclusions can be reached regarding the quality and performance of a company's products.

Table I lists other international test methods that are equivalent to the ASTM test methods. No claim is being made that all details in ASTM and non-ASTM standards are exactly the same. However, it is expected that if properly followed, both sets should give equivalent results. In an increasingly global marketplace, it is important to be cognizant of such equivalency. Much of this information on the equivalent test methods has been extracted from a compilation that has been prepared by Mr. Tim Berryman and published by Institute of Petroleum, London. Although at one time leading European national standards organizations such as IP, DIN, and AFNOR produced their own independent standards, lately they are being integrated with the ISO (or more specifically EN-ISO) series standards. The reason for this is that there is a European Union legal requirement to publish all EN standards as the national standards and to withdraw all existing conflicting standards. The vast majority of EN standards are actually implementation of the correspond-

ing ISO standards. Table I retains the original designations of the European national bodies with which most people are familiar, rather than the new common EN-ISO designations. Again, it is not claimed that the alternate methods are exactly equivalent, but it should be remembered that they may be technically equivalent or technically related. The readers are encouraged to consult the IP publication of Mr. Berryman's compilation for better understanding of the relationship among the test methods. My thanks to Mr. Berryman for permission to include his data here.

All technical information included in this book is based on the Year 2000 editions of the *Annual Book of ASTM Standards* [2]. We plan to update this manual at some frequency depending on how many new or significantly revised test method standards are issued by ASTM Committee D02 on Petroleum Products and Lubricants. If you notice any errors or omissions, please let us know. We will correct them in future editions.

References

[1] Nadkarni, R. A., editor—"Modern Instrumental Methods of Analysis of Petroleum Products and Lubricants," *ASTM STP 1109,* ASTM, 1991.
[2] *Annual Book of ASTM Standards,* Volumes 05.01, 05.02, 05.03, and 05.04.
[3] Dyroff, G. V., ed. *Manual on Significance of Tests for Petroleum Products,* 5th ed., ASTM, West Conshohocken, PA, MNL 1, 1993.

Explanation of Terms

Accuracy—The accuracy of a test is a measure of how close the test result will be to the true value of the property being measured. As such the accuracy can be expressed as the bias between the test result and the true value. However, the absolute accuracy can only be established if the true value is known.
AFNOR—Association Francaise de Normalisation (Paris).
ASTM—American Society for Testing and Materials (U.S.A.).
DIN—Deutsche Institut Fur Normung (Germany).
IP—Institute of Petroleum (U.K.).
ISO—International Organization for Standardization (Switzerland).
JIS—Japan Industrial Standards (Tokyo).
Precision—The precision of a test method is defined in terms of the variability between test results obtained on the same material, using a specific test method. The precision of a test is usually unrelated to its accuracy. The results may be precise, but not necessarily accurate. Figures 1 to 4[1] depict in a bull's eye analogy the relation between precision and accuracy. Ideal condition would be most precise and most accurate results. Precision is expressed as repeatability and reproducibility.
Repeatability—The "within-laboratory precision" refers to the precision of a test method when the results are obtained by the same operator in the same laboratory using the same apparatus.

Repeatability or repeatability interval of a test (indicated with the letter "*r*") is defined as the maximum permissible difference due to test error between two results obtained on the same material in the same laboratory.

$$r = 2.77 \times \text{standard deviation of test}$$

Most commonly this repeatability interval (r) is statistically defined at the 95% probability level, meaning that, even in normal conditions, differences between two test results are unlikely to exceed this repeatability interval more than five times in a hundred.
Reproducibility—The "between-laboratory precision" is defined in terms of the variability between test results obtained on the aliquots of the same homogeneous material in different laboratories using the same test method.

The term reproducibility or reproducibility interval (designated as "*R*") is completely analogous to the term repeatability. Only in this case, it is the maximum permissible difference between two results obtained on the same material but now in different laboratories. The statistical definition of reproducibility is along the same lines as above. Therefore differences between two or more laboratories are unlikely to exceed the reproducibility interval more than five times in a hundred.

$$R = 2.77 \times \text{standard deviation of test}$$

Reproducibility is generally higher than repeatability by a factor of 2 to 4.

The repeatability and reproducibility values, have very important implications in today's quality conscious market. As the demand for clear product specifications, and hence control over product consistency grows, it is meaningless to establish product specifications that are more restrictive than the reproducibility/repeatability values of the specification test methods.

[1] Committee on Standards, "Precision and Bias" *ASTM Standardization News,* ASTM, January 1985, p. 45.

TABLE I—Test Method Equivalence.[a]

Analysis	ASTM	IP	ISO	DIN	JIS	AFNOR
Tag flash point	D 56			51411	K 2580	M07-003
Distillation	D 86	123	3405	51751	K 2254	M07-002
Melting point	D 87	55	3841	51570		T60-114
Cleveland Open Cup flash point	D 92	36	2592	51376	K 2265	T60-118
Pensky-Martens flash point	D 93	34	2719	51758	K 2265	M07-019
Saponification number	D 94	136	6293	51559	K 2503	T60-110
Water distillation	D 95	74	3733	51582	K 2275	T60-113
Pour point	D 97	15	3016	51597	K 2269	T60-105
Drop melting point	D 127	133	6244			T60-121
Sulfur, bomb method	D 129	61		51577		T60-109
Copper corrosion	D 130	154	2160	51759	K 2513	M07-015
Saybolt color	D 156			51411	K 2580	M07-003
Conradson carbon residue	D 189	13	6615	51551	K 2270	T60-116
Cone penetration	D 217	50	2137			T60-132
Specific energy, bomb method	D 240	12		51900		M07-030
Reid vapor pressure	D 323	69	3007	51754	K 2258	M41-007
Gum in fuels	D 381	131	6246	51784	K 2261	M07-004
Kinematic viscosity	D 445	71-1	3104	51562	K 2283	T60-100
Kinematic viscometer calibration	D 446	71-2	3105	51562		
Sediment	D 473	53	3735	51789		M07-063
Ash	D 482	4	6245		K 2272	M07-045
Ramsbottom carbon residue	D 524	14	4262			T60-117
Oxidation stability	D 525	40	7536	51780		M07-012
Dropping point	D 566	132	2176			T60-102
Aniline point	D 611	2	2977	51775		M07-021
Ignition quality	D 613	41	5165		K 2280	M07-035
Neutral number	D 664	177	6619		K 2501	
Rust	D 665	135	7120	51585	K 2510	T60-151
Wax oil	D 721	158	2908			T60-120
Oxidation stability	D 873	138				M07-013
Sulfated ash	D 874	163	3987	51575	K 2272	T60-143
Foam	D 892	146	6247	51566	K 2518	T60-129
Cone penetration	D 937	179		51580		T60-119
Congealing point	D 938	76	2207			T60-128
Oxidation stability	D 942	142		51808		
Oxidation characteristics of inhibited mineral oils	D 943		4263			
Neutral number	D 974	139	6618	51558T1	K 2501	T60-112
Water reaction	D 1094	289	6250	51415		M07-050
Bromine number	D 1159	130	3839			
Distillation characteristics at reduced pressures	D 1160		6616			
Refractive index	D 1218		5661	51423T2		
Water washout	D 1264	215	11009	51807T2		
Sulfur by lamp	D 1266	107				M07-031
Liquefied petroleum gases (LPG) vapor pressure	D 1267	161	4256	51616		
Density	D 1298	160	3675	51757H	K 2249H	T60-101
Hydrocarbon types	D 1319	156	3837	51791	K 2536	M07-024
Needle penetration	D 1321	49		51579		T66-004
Smoke point	D 1322	57	3014	51406	K 2537	M07-028
Water separability	D 1401	412	6614			T60-125
Cone penetration	D 1403	310				T60-140
Color, ASTM	D 1500	196	2049	51578	K 2580	T60-104
Vanadium in fuel oils	D 1548					M07-027
Sulfur by high temperature	D 1552					M07-025
Density	D 1657	235	3993			M41-008
Karl Fischer water	D 1744		6296			T60-154
Sediment and water	D 1796	75	3734	51793		M07-020
Copper corrosion in LPG	D 1838	411	6251			M41-010
Gas chromatography (GC) analysis of LPG	D 2163	264	7941	51619		M41-013
Dropping point lubricating grease	D 2265		6299			
Four ball method	D 2266	239	11008	51350		
Viscosity index calculations	D 2270	226	2909			T60-136
Oxidation stability	D 2274	388	12205			
Freezing point	D 2386	16	3013	51421	K 2276	M07-048
H_2S in LPG	D 2420	401	8819	51855T3		M41-011
Cloud point	D 2500	219	3015	51597	K 2269	T60-105
Lead, volumetric	D 2547	248	2083			M07-014
Extreme pressure properties	D 2596		11008			

TABLE I—Test Method Equivalence.[a] (*Continued*)

Analysis	ASTM	IP	ISO	DIN	JIS	AFNOR
LPG properties	D 2598	432	8973			
Cold cranking simulator	D 2602	350		51377		
Sulfur by X-ray fluorescence	D 2622			51400T6	K 2541	
Electrical conductivity	D 2624	274	6297	51412T2		
Timken	D 2782	240		51434		
Suilfur by Wickbold combustion	D 2785	243	4260			T60-142
Boiling range by GC	D 2887		3924			
Distillation characteristics with 15-plate column	D 2892		8708			
Base number	D 2896	276	3771		K 2501	
Brookfield viscosity	D 2983	267	9262			T42-011
Sulfur by oxidative microcoulometry	D 3120		16591			
Mercaptan sulfur	D 3227	342	3012		K 2276	M07-022
Lead by atomic absorption spectroscopy (AAS)	D 3237	428				
JFTOT	D 3241	323	6249		K 2276B	M07-051
Acidity of aviation turbine fuel (ATF)	D 3242	354		51558T3		
Sulfur by oxidative microcoulometry	D 3246	373				M07-052
Acid numbers, semi-micro method	D 3339		7537			
Lead by iodine chloride	D 3341		3830			
Metals by atomic absorption (AAS)	D 3605	413	8691	51790T3		
Benzene by GC	D 3606	425				
Sulfonates	D 3712	369				
Seta flash point	D 3828	303	3679			
Water by distillation	D 4006	358				
Water and sediment	D 4007	359				
Alkyl nitrate	D 4046	430	13759			
Phosphorus by colorimetry	D 4047	149	4265			
Density, digital	D 4052	365	12185	51757D	K 2249D	T60-172
Benzene by infrared spectroscopy (IR)	D 4053	429				
Sulfur by ND-XRF	D 4294	336	8754			M07-053
Karl Fischer water	D 4377	356				
Aniline point	D 4529	381	3648			
Microcarbon residue	D 4530	398	10370			
Metals by AAS	D 4628	308		51391T1		
Nitrogen by chemiluminescence detection	D 4629	379				M07-058
Base number	D 4739	417	6619		K 2501	
Sediment	D 4870	375	10307			
Metals by wavelength-dispersive X-ray fluorescence (WDXRF)	D 4927	407		51391T2		
Karl Fischer water	D 4928	386	10337			
Doctor test	D 4952	30	5275			
Aluminum and silicon in fuels	D 5184	377	10478	51416		
Vapor pressure	D 5191	394				M07-079
Shear stability index	D 5275	294		51382		
Oxygenates	D 5599	408				
Noack volatility	D 5800	421		51581		
Freezing point	D 5901	434				
Auto-freeze point	D 5972	435				
Particulate matter	D 6217	415	15167			
Cold filter plugging	D 6371	309	116			
Friction and wear	D 6425			51834		
Aromatic hydrocarbon	D 6379	436				

[a] For further information, see the Introduction.

TABLE II—Alphanumeric Index Reference to ASTM Standards by Designation Number.

TABLE II—Alphanumeric Index Reference to ASTM Standards by Designation Number. (*Continued*)

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D 1657	Density or Relative Density of Light Hydrocarbons by Pressure Thermohydrometer (Discontinued 1998)	99
D 1662	Active Sulfur in Cutting Oils	18
D 1740	Luminometer Numbers of Aviation Turbine Fuels	210
D 1743	Corrosion Preventive Properties of Lubricating Greases	92
D 1744	Water in Liquid Petroleum Products by Karl Fischer Reagent	370
D 1747	Refractive Index of Viscous Materials	279
D 1796	Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)	373
D 1831	Roll Stability of Lubricating Grease	280
D 1832	Peroxide Number of Petroleum Wax	262
D 1833	Odor of Petroleum Wax	241
D 1839	Amyl Nitrate in Diesel Fuels	23
D 1840	Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet-Spectrophotometry	235
D 1957	Hydroxyl Value of Fatty Oils and Acids	195
D 2001	Dependanization of Gasoline and Napthas	107
D 2007	Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method	175
D 2008	Ultraviolet Absorbance and Absorptivity of Petroleum Products	335
D 2068	Filter Blocking Tendency of Distillate Fuel Oils	138
D 2070	Thermal Stability of Hydraulic Oils	328
D 2078	Iodine Value of Fatty Quaternary Ammonium Chlorides	198
D 2161	Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity	355
D 2265	Dropping Point of Lubricating Grease Over Wide Temperature Range	119
D 2266	Wear Preventing Characteristics of Lubricating Grease (Four-Ball Method)	386
D 2270	Calculation of Viscosity Index from Kinematic Viscosity at 40 and 100°C	351
D 2273	Trace Sediment in Lubricating Oils	287
D 2274	Oxidation Stability of Distillate Fuel Oil (Accelerated Method)	246
D 2276	Particulate Contamination in Aviation Fuel by Line Sampling	258
D 2319	Softening Point of Pitch (Cube-in-Air Method)	300
D 2320	Density (Specific Gravity) of Solid Pitch (Pycnometer Method)	102
D 2386	Freezing Point of Aviation Fuels	152
D 2392	Color of Dyed Aviation Gasolines	75
D 2415	Ash in Coal Tar and Pitch	37
D 2416	Coking Value of Tar and Pitch (Modified Conradson)	86
D 2420	Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method)	191
D 2425	Hydrocarbon Types in Middle Distillates by Mass Spectrometry	183
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D 2502	Molecular Weight (Relative Molecular Mass) of Petroleum Oils from Viscosity Measurements	230
D 2503	Relative Molecular Mass (Molecular Weight) of Hydrocarbons by Thermoelectric Measurement of Vapor Pressure	229
D 2505	Ethylene, Other Hydrocarbons, and Carbon Dioxide in High-Purity Ethylene by Gas Chromatography	179
D 2509	Load-Carrying Capacity of Lubricating Grease (Timken Method)	205
D 2510	Adhesion of Solid Film Lubricants	20
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D 2532	Viscosity and Viscosity Change After Standing at Low Temperature of Aircraft Turbine Lubricants	352
D 2533	Vapor Liquid Ratio of Spark-Ignition Engine Fuels	338
D 2549	Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography	176
D 2569	Distillation of Pitch	116
D 2595	Evaporation Loss of Lubricating Greases	127
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D 2603	Sonic Shear Stability of Polymer-Containing Oils	294
D 2619	Hydrolytic Stability of Hydraulic Fluids (Beverage Bottle Method)	190
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D 2624	Electrical Conductivity of Aviation and Distillate Fuels	121
D 2638	Real Density of Calcined Petroleum Coke by Helium Pycnometer	94
D 2649	Corrosion Characteristics of Solid Film Lubricants	90
D 2669	Apparent Viscosity of Petroleum Waxes Compounded with Additives (Hot Melts)	348
D 2709	Water and Sediment in Distillate Fuels by Centrifuge	290
D 2710	Bromine Index of Petroleum Hydrocarbons by Electrometric Titration	57
D 2711	Demulsibility Characteristics of Lubricating Oils	93
D 2717	Thermal Conductivity of Liquids	326
D 2782	Extreme-Pressure Properties of Lubricating Fuels (Timken Method)	133
D 2783	Extreme-Pressure Properties of Lubricating Fuels (Four Ball Method)	134
D 2784	Sulfur in Liquified Petroleum Gases (Oxy-Hydrogen Burner or Lamp)	316
D 2786	Hydrocarbon Types Analysis of Gas-Oil Saturate Fractions by High Ionizing Voltage Mass Spectrometry	180
D 2789	Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry	184
D 2878	Apparent Vapor Pressures and Molecular Weights of Lubricating Oils	228
D 2882	Indicating the Wear Characteristics of Petroleum and Non-Petroleum Hydraulic Fluids in a Constant Volume Vane Pump	387
D 2887	Boiling Range Distribution of Petroleum Fractions by Gas Chromatography	59
D 2890	Liquid Heat Capacity of Petroleum Distillate Fuels	172
D 2892	Distillation of Crude Petroleum (15-Theoretical Plate Column)	110
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D 2896	Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration	45
D 2981	Wear Life of Solid Film Lubricants in Oscillating Motion	388
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D 3120	Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry	318
D 3227	Mercaptan Sulfur in Gasoline, Kerosene, Aviation Turbine, and Distillate Fuels (Potentiometric Method)	214
D 3228	Total Nitrogen in Lubricating Oils and Fuel Oils by Modified Kjeldahl Method	240
D 3230	Salts in Crude Oil (Electrometric Method)	283
D 3231	Phosphorus in Gasoline	265
D 3233	Extreme Pressure Properties of Fluid Lubricants (Falex Pin and Vee Block Methods)	132
D 3235	Solvent Extractable in Petroleum Waxes	137
D 3236	Apparent Viscosity of Hot Melt Adhesives and Coating Materials	347
D 3237	Lead in Gasoline by Atomic Absorption Spectroscopy	199
D 3239	Aromatic Types Analysis of Gas-Oil Aromatic Fractions by High Ionizing Voltage Mass Spectrometry	181
D 3240	Undissolved Water in Aviation Turbine Fuels	381
D 3241	Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT Procedure)	327
D 3242	Acidity in Aviation Turbine Fuel	16
D 3245	Pumpability of Industrial Fuel Oils	276
D 3246	Sulfur in Petroleum Gas by Oxidative Microcoulometry	319
D 3338	Net Heat of Combustion of Aviation Fuels	165
D 3339	Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration	14
D 3340	Lithium and Sodium in Lubricating Greases by Flame Photometry	204
D 3341	Lead in Gasoline—Iodine Monochloride Method	200
D 3343	Hydrogen Content of Aviation Fuels	186
D 3348	Rapid Field Test for Trace Lead in Unleaded Gasoline (Colorimetric Method)	202
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D 3461	Softening Point of Asphalt and Pitch (Mettler Cup-and-Ball Method)	298
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D 3523	Spontaneous Heating Values of Liquids and Solids (Differential Mackey Test)	169
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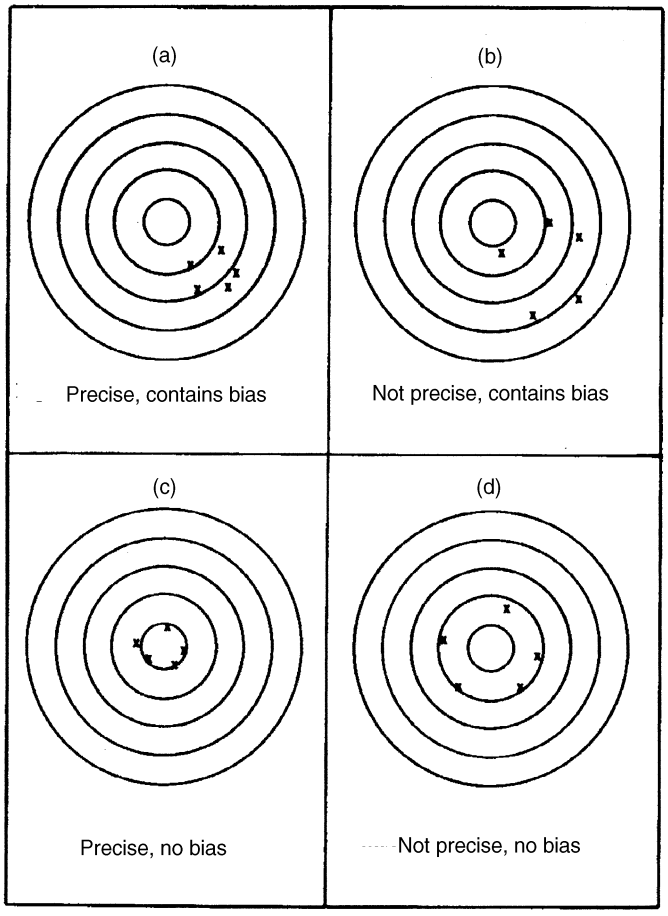

FIG. 1—Bull's eye analogy.