

# Appendix

---

Table

**A-1. Critical Values of the F Test (95% confidence values)**

$df_D$	$df_N$									
	1	2	3	4	5	6	7	8	9	10
1	648	800	864	900	922	937	948	956	963	968
2	38.5	39.0	39.2	39.3	39.3	39.4	39.4	39.4	39.4	39.4
3	17.4	16.0	15.4	15.1	14.9	14.7	14.6	14.5	14.5	14.4
4	12.2	10.6	9.98	9.60	9.36	9.20	9.07	8.98	8.90	8.84
5	10.0	8.43	7.76	7.39	7.14	6.98	6.85	6.75	6.68	6.62
6	8.81	7.26	6.60	6.23	5.99	5.82	5.70	5.60	5.52	5.46
7	8.07	6.54	5.89	5.52	5.28	5.12	4.99	4.90	4.82	4.76
8	7.57	6.06	5.42	5.05	4.82	4.65	4.53	4.43	4.36	4.29
9	7.21	5.71	5.08	4.72	4.48	4.32	4.20	4.10	4.03	3.96
10	6.94	5.46	4.82	4.47	4.24	4.07	3.95	3.85	3.78	3.72

Note:  $df_N$  is the degrees of freedom in the numerator, and  $df_D$  is the degrees of freedom in the denominator of F.

Table

**A-2. Critical Values of the F Test (99% confidence values)**

$df_D$	$df_N$								
	1	2	3	4	5	6	7	8	9
1	16,211	20,000	21,615	22,500	23,056	23,437	23,715	23,925	24,091
2	198.5	199.0	199.2	199.3	199.3	199.3	199.4	199.4	199.4
3	55.5	49.8	47.5	46.2	45.4	44.8	44.4	44.1	43.9
4	31.3	26.3	24.3	22.2	22.5	22.0	21.6	21.4	21.1
5	22.8	18.3	16.5	15.6	14.9	14.5	14.2	14.0	13.8
6	18.6	14.5	12.9	12.0	11.5	11.1	10.8	10.6	10.4
7	16.2	12.4	10.9	10.0	9.52	9.15	8.89	8.67	8.51
8	14.7	11.0	9.60	8.80	8.30	7.95	7.69	7.50	7.34
9	13.6	10.1	8.72	7.96	7.47	7.13	6.88	6.69	6.54
10	12.8	9.43	8.08	7.34	6.87	6.54	6.30	6.12	5.97

Note:  $df_N$  is the degrees of freedom in the numerator, and  $df_D$  is the degrees of freedom in the denominator of F.

# Additional Readings

---

## Journal Articles

- Bankston, D. 1984. Elimination of Bias in Sample Selection for Chemical Analysis, *American Laboratory*, pp. 43–48 (December 1984).
- Boomer, B., T. Dux, and D. March. Sampling Surveys of Hazardous Waste, *Journal of Air Pollution Control and Waste Management*, 38:1426–1432 (1988).
- Dols, T., and B. Armbrrecht. Assessment of Analytical Method Performance Characteristics, *Journal of the AOAC*, 60:940–945 (1977).
- Dux, J. P. Quality Assurance in the Analytical Laboratory, *American Laboratory*, pp. 212–216 (July 1983).
- Glacer, J. L. Trace Analysis for Wastewater, *Environmental Science and Technology*, 15:1426–1455 (1981).
- Horwitz, W., L. R. Kampes, and K. W. Boyer. Quality Assurance in the Analysis of Foods for Trace Constituents, *Journal of the Association of Official Analytical Chemists*, 63:1344–1354 (1980).
- Hubaux, A., and G. Vos. Precision and Detection Limits for Linear Calibration Curves, *Analytical Chemistry*, 42:849–855 (1970).

- Kanzelmyer, J. H. Quality Control for Analytical Methods, *ASTM Standardization News*, pp. 25–28 (October 1977).
- Keith, L. H. et al. Principles of Environmental Analysis, *Analytical Chemistry*, 55:2210–2218 (1983).
- Kirchmer, C. J. Quality Control in Water Analysis, *Environmental Science and Technology*, 17:174A–184A (1983).
- Kratchovil, B., D. Wallace, and J. Taylor. Sampling for Chemical Analysis, *Chemical Reviews*, 56:113R (1984).
- MacDougall, D. et al. Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry, *Analytical Chemistry*, 52:2242–2249 (1980).
- Mandel, J. The Analysis of Interlaboratory Test Data, *ASTM Standardization News*, pp. 17–20 (1977).
- Mandel, J., and F. C. Linnig. Study of Accuracy in Chemical Analyses Using Linear Calibration Curves, *Analytical Chemistry*, 29:743–749 (1959).
- Provost, L. P., and R. S. Elder. Interpretation of Percent Recovery Data, *American Laboratory*, pp. 57–63 (1983).
- Saltzman, B. E., D. W. Yeager, and B. G. Meiners. Reproducibility and Quality Control in the Analysis of Biological Samples for Lead and Mercury, *American Industrial Hygiene Association Journal*, 44:163–167 (1983).
- Schwartz, L. M. Nonlinear Calibration, *Analytical Chemistry*, 49:2062–2068 (1977).
- Youden, W. Accuracy of Analytical Procedures, *Journal of the Association of Official Analytical Chemists*, 45:169–173 (1962).

## Books

- Beizer, P. *The Frozen Keyboard: Living with Bad Software*, Blue Ridge Summit, Pa.: TAB Books, Inc., 1988.
- Dessy, R. *The Electronic Laboratory*, Washington, D.C.: American Chemical Society, 1985.
- Dowdey, S., and S. Wearden. *Statistics for Research*. New York: John Wiley and Sons, 1983.
- Fites, P., P. Johnston, and M. Kratz. *The Computer Virus Crisis*, New York: Van Nostrand Reinhold, 1989.

- Garfield, F. M. *Quality Assurance Principles for Analytical Laboratories*, Arlington, Va.: Association of Official Analytical Chemists, 1984.
- Hurst, W., and J. Mortimer. *Laboratory Robotics*, New York: VCH Publishers, 1987.
- Inhorn, S. L. (ed.) *Quality Assurance Practices for Health Laboratories*, Washington, D.C.: American Public Health Association, 1978.
- Kanare, H. W. *Writing the Laboratory Notebook*, Washington, D.C.: American Chemical Society, 1985.
- Mahaffey, R. *LIMS: Applied Information Technology for the Laboratory*, New York: Van Nostrand Reinhold, 1990.
- Schulmeyer, G., and J. McManus (eds.) *Handbook of Software Quality Assurance*, New York: Van Nostrand Reinhold, 1987.
- Taylor, J. *Quality Assurance of Chemical Measurements*, Chelsea, Mich.: Lewis Publishers, 1988.
- Youden, W. J., and E. H. Steiner. *Statistical Manual of the AOAC*, Washington, D.C.: Association of Official Analytical Chemists, 1975.

# INDEX

---

- accreditation, 181–189
  - by discipline, 182
  - by field of testing, 182
  - by-product/standard, 182
  - cost and benefits of, 188
  - criteria for, 184
  - process 185
- ACIL. *See* American Council of Independent Laboratories
- A2LA. *See* American Association for Laboratory Accreditation
- American Association of Feed Control Operators, 45
- American Association for Laboratory Accreditation, 182
- American Council of Independent Laboratories, 182
- American Industrial Hygiene Association, 183
- American Oil Chemists Society, 45
- American Society for Testing and Materials, 92
- analytical data, accountability of, 7
  - importance of, 2
  - traceability of, 7
- analytical methods, 59–77
  - authorization of, 75–77
    - form for, 76
  - comparison of, 67–69
  - evaluation, 67–71
  - general principles, 60
  - in-house developed, 62
  - methods manual, 102
  - official, 61
  - ruggedness test of, 69–71
  - sources of, 60–62
  - standard, 61
  - validation studies, 62–69
    - example of, 62–67
  - writing, 71–75
    - format for, 71–75
- analytical reports, 112–114
- analytical system, definition of, 6
- ANOVA, 23
- archives, area for, 160
- archivist, 160
- auditing, 145–153
  - assessor's checklist, typical, 151–153
  - external audits, 148–153

- auditing (*cont.*)
  - external audits (*cont.*)
    - method audits, 150
    - performance audits, 150
    - system audits, 149
    - testing technology audits, 149
  - internal audits, 146–148
    - method audits, 148
    - performance audits, 148
    - system audits, 146–148
  - types of audits, 145–146
- average, arithmetic, 15
- Beizer, B., 168, 174
- blind samples, 42–44
  - and blunders, 42
  - definition, 42
- blunders, 11–12
- calibration, 80–87
  - analytical balances, 84
  - atomic absorption
    - spectrophotometers, 86
  - conductivity meters, 86
  - frequency of, 83
  - furnaces, 85
  - gas chromatographs, 86
  - glassware, 84
  - high performance liquid chromatographs, 86
  - infrared spectrophotometers, 86
  - linear calibration curves, 81–83
  - methods, written, 107
  - notebooks, 107
  - ovens, 84
  - pH meters, 85
  - ultraviolet/visible
    - spectrophotometers, 85
- certification, 181
- chain of custody, 106
- check standards, characteristics of, 36–37
- computer hardware, 161–162
- computer viruses, 171
- confidence limit, 20
- confidence range, 20
- control charting, 31–42
  - R charts, 39
  - spiked sample, 38–39
  - types of, 32
  - X charts, 33
- control limits, 32
- data acquisition, 92–101
  - direct computer, 100–101
  - electronic, 100
  - manual, 97–99
- degrees of freedom, 16
- deviation, average, 15
- deviation, standard, 16
  - equations for, 16
  - of the mean, 22
  - relative, 17
- distribution, Gaussian, 18
- distribution, normal, 18–20
  - equation of 19
- documentation, equipment maintenance, 107
- documentation, instrument calibration, 107
- documents, quality assurance, filing, 114–116
- Elder, R. S., 46
- electrical service requirements, 156
- emergency power source, 156
- EPA, 183
- equipment maintenance, 87–90
- error, 11–13
  - categories of, 11–13
  - due to bias, 12
  - random, 12
    - distribution of, 13
  - systematic, 12
- F-factor, 65
- F tables, 196–197
- F test, 23–24



- facilities for quality assurance, 155–160  
 archives, 160  
 general requirements, 155–157  
 glassware cleaning, 159  
 sample disposal, 159  
 sample receipt, 157–158  
 sample storage, 158–159
- Fites, P., 174
- glassware, maintenance, 89–90
- GLPs. *See* Good Laboratory Practices
- Good Laboratory Practices, 176–179
- Gossett, W. S., 20
- Halon fire extinguishers, 156
- Hess, E. H., 189
- information  
 analytical data as, 1  
 characteristics of, 1–2  
 society, 1
- instrument and equipment, general  
 principles for Q.A., 80
- instrument room, separate, 156
- International Laboratory Accreditation Conference, 184
- Johnston, P., 174
- Kanzelmeyer, J. H., 10
- Kirchner, C. J., 10
- Kratz, M., 174
- laboratory accreditation, 181–189
- Laboratory Information Management System, 162–172  
 calculation of analytical results, 160  
 control charting, 166  
 documentation of, 169  
 management of, 168–172  
 reporting results, 166
- Laboratory Information Management System (*cont.*)  
 results verification, 165  
 sample log-in, 163  
 sample status, 165  
 security and, 170–171  
 selecting a, 168  
 software quality assurance and, 169–170
- LCL. *See* control limits
- least squares straight line, 64–65
- LIMS. *See* Laboratory Information Management System
- Linnig, F. J., 65, 77
- LWL. *See* warning limits
- Mahaffey, R., 163, 168, 174
- maintenance notebooks, 107
- maintenance, preventive, 108
- Mandel, J., 65, 77
- McManus, J., 170, 174
- mean, 15
- mean, geometric, 15
- means, comparison of, 27–29
- median, 15
- mode, 15
- Mortimer, J. W., 174
- National Institute for Standards and Technology, 90
- National Voluntary Laboratory Accreditation Program, 182–183
- notebooks, 98–100  
 control of, 98  
 data entry in, 99
- performance evaluation samples, 44–46  
 and laboratory accreditation, 44
- Prontius, P. E., 46
- Provost, L. P., 46
- quality, and accuracy, 4
- quality, definition of, 3–4

- quality assurance, 7–8
  - definition of, 7
  - documentation, general
    - principles of, 96
    - attributability, 96
    - permanence, 96
    - security, 97
  - irregularity report form, 138
  - management commitment to, 117
  - manual, suggested outline, 141–144
    - writing the, 140–144
  - motivating personnel, 126–127
  - objectives, 7
  - responsibility for, 123–126
    - bench analysts, 126
    - quality assurance officer, 125
    - senior management, 123
    - supervisory personnel, 124
    - support personnel, 126
  - unit (QAU), 178
- quality assurance officer, 118–125
  - duties and responsibilities, 121–122
  - education and experience, 119
  - general characteristics, 120
  - knowledge of statistics, 120
  - place in organization, 122
- quality assurance problems, reporting, 137–139
- quality assurance program, 129–144
  - consolidating the, 136–137
  - defining the, 129
  - mandated, 175–181
  - monitoring and evaluating, 137
- quality control, and the analytical system, 6
- quality control, definition of, 5
- range, 15, 25
  - of duplicates, distribution of, 39
- reagents
  - contamination of, 91
  - grades, 91
  - labeling, 91, 109
- reagents (*cont.*)
  - purchasing, 108
- reagent solutions
  - labeling, 91, 109
  - preparation notebook, 109
- records, personnel training, 110–112
  - non-degreed personnel, 111
  - professional personnel, 110
  - responsibility for, 111
- record retention time, 160
- robotics, laboratory, 172–174
- sample
  - handling in the laboratory, 55
  - holding time, 54
  - labeling, 103
  - log-in, 104–106
  - preservation, 54–55
- samples
  - contamination of, 55
  - forensic, 106
- sampling
  - documentation, 56–57
  - matrix, homogeneity of, 50
  - methods manual, 103
  - nature of matrix, 50
  - plans, 48
    - objectives, 49
  - protocol samples, 51
  - quality assurance and, 47–57
  - random samples, 51
  - representative samples, 51
  - selective samples, 51
  - statistical considerations, 51–53
  - techniques, 53–54
  - type of sample, 51
- Schulmeyer, G., 170, 174
- Schumaker, D., 116
- Shewhart, W. A., 32, 46
- SOP. *See* standard operating procedures
- standard operating procedures,
  - 131–135
  - format, 132–133
  - suggested topics, 133–135

- standards
  - handling, 90
  - primary, 90
  - reference materials (SRMs), 90
  - secondary, 90
- statistics, 13–19
  - quality control and, 31–46
- Steiner, E. H., 39, 46, 77
- student, 21
- Taylor, J. K., 57
- technical complaints, handling,  
139–140
- t*-factor, 21
- UCL. *See* Control limits
- uninterruptable power supply, 156
- U.S. Army Toxic and Hazardous  
Waste Materials Agency, 180
- U.S. Environmental Protection  
Administration (EPA), 176
- U.S. Food and Drug Administration  
(FDA), 176
- variance, 23–29
  - analysis of, 23
  - definition of, 16
  - from duplicate analyses, 25
  - pooling of, 24
- variation, coefficient of, 17
- voltage, surge protection, 156
- warning limits, 32
- water
  - purification of, 92
  - quality of, 92
- Youden, W. J., 39, 46, 77