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(e) The concentration of each test atmosphere shall be established and/or verified before or during each series of tests. Samples for verifying test concentrations shall be collected from the test atmosphere delivery system as close as possible to the sample intake port of the test analyzer.

(f) The accuracy of all flow measurements used to calculate test atmosphere concentrations shall be documented and referenced to a primary standard (such as a spirometer, bubble meter, etc.). Any corrections shall be clearly shown. All flow measurements given in volume units shall be standardized to 25 °C. and 760 mm Hg.

(g) Schematic drawings and other information showing complete procedural details of the test atmosphere generation, verification, and delivery system shall be provided. All pertinent calculations shall be clearly indicated.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975]

§ 53.23 Test procedures.

(a) *Range*—(1) *Technical definition.* Nominal minimum and maximum concentrations which a method is capable of measuring.

NOTE: The nominal range is specified at the lower and upper range limits in concentration units, for example, 0–0.5 p/m.

(2) *Test procedure.* Submit a suitable calibration curve, as specified in § 53.21(b), showing the test analyzer's response over at least 95 percent of the required range.

NOTE: A single calibration curve will normally suffice.

(b) *Noise*—(1) *Technical definition.* Spontaneous, short duration deviations in output, about the mean output, which are not caused by input concentration changes. Noise is determined as the standard deviation about the mean and is expressed in concentration units.

(2) *Test procedure.* (i) Allow sufficient time for the test analyzer to warm up and stabilize. Determine at two concentrations, first using zero air and then a pollutant test gas concentration as indicated below. The noise specification in table B–1 shall apply to both of these tests.

(ii) Connect an integrating-type digital meter (DM) suitable for the test analyzer's output and accurate to three significant digits, to measure the analyzer's output signal.

NOTE: Use of a chart recorder in addition to the DM is optional.

(iii) Measure zero air for 60 minutes. During this 60-minute interval, record twenty-five (25) readings at 2-minute intervals. (See Figure B–2 in appendix A.)

(iv) Convert each DM reading to concentration units (p/m) by reference to the test analyzer's calibration curve as determined in § 53.21(b). Label the converted DM readings r_1, r_2, r_3 . . . r_i . . . r_{25}.

(v) Calculate the standard deviation, S, as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{25}(r_i)^2 - \frac{1}{25}\left(\sum_{i=1}^{25} r_i\right)^2}{24}} \; (p/m)$$

where i indicates the i-th DM reading in ppm.

(vi) Let S at 0 ppm be identified as S_o; compare S_o to the noise specification given in table B–1.

(vii) Repeat steps (iii) through (vi) of this section using a pollutant test atmosphere concentration of 80±5 percent of the upper range limit (URL) instead of zero gas, and let S at 80 percent of the URL be identified as S_{80}. Compare S_{80} to the noise specification given in table B–1.

(viii) Both S_0 and S_{80} must be less than or equal to the specification for noise to pass the test for the noise parameter.

(c) *Lower detectable limit*—(1) *Technical definition.* The minimum pollutant concentration which produces a signal of twice the noise level.

(2) *Test procedure.* (i) Allow sufficient time for the test analyzer to warm up and stabilize. Measure zero air and record the stable reading in ppm as B_Z. (See Figure B–3 in appendix A.)

(ii) Generate and measure a pollutant test atmosphere concentration equal to the value for the lower detectable limit specified in table B–1.

NOTE: If necessary, the test atmosphere concentration may be generated or verified at a higher concentration, then accurately

diluted with zero air to the final required concentration.

(iii) Record the test analyzer's stable indicated reading, in ppm, as B_L.

(iv) Determine the Lower Detectable Limit (LDL) as LDL = $B_L - B_Z$. Compare this LDL value with the noise level, S_0, determined in §53.23(b), for 0 concentration test atmosphere. LDL must be equal to or higher than $2S_0$ to pass this test.

(d) *Interference equivalent*—(1) *Technical definition.* Positive or negative response caused by a substance other than the one being measured.

(2) *Test procedure.* The test analyzer shall be tested for all substances likely to cause a detectable response. The test analyzer shall be challenged, in turn, with each interfering agent specified in table B–3. In the event that there are substances likely to cause a significant interference which have not been specified in table B–3, these substances shall be tested at a concentration substantially higher than that normally found in the ambient air. The interference may be either positive or negative, depending on whether the test analyzer's response is increased or decreased by the presence of the interferent. Interference equivalents shall be determined by mixing each interferent, one at a time, with the pollutant at the concentrations specified in table B–3, and comparing the test analyzer's response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between an interferent and the pollutant are designated by footnote 3 in table B–3. In these cases, the interference equivalent shall be determined in the absence of the pollutant.

(i) Allow sufficient time for warm-up and stabilization of the test analyzer.

(ii) For a candidate method using a prefilter or scrubber based upon a chemical reaction to derive part of its specificity, and which requires periodic service or maintenance, the test analyzer shall be ''conditioned'' prior to each interference test as follows:

TABLE B–3—INTERFERANT TEST CONCENTRATION,[1] PARTS PER MILLION

Pollutant	Analyzer type[2]	Hydrochloric acid	Ammonia	Hydrogen sulfide	Sulfur dioxide	Nitrogen dioxide	Nitric oxide	Carbon dioxide	Ethylene	Ozone	M-xylene	Water vapor	Carbon monoxide	Methane	Ethane
SO_2	Flame photometric (FPD)			0.1	[1] 0.14			750				[3] 20,000	50		
SO_2	Gas chromatography (FPD).			.1	[4] .14			750				[3] 20,000	50		
SO_2	Spectrophotometric-wet chemical (pararosaniline reaction).	0.2	[3] 0.1	.1	[4] .14	0.5		750		0.5					
SO_2	Electrochemical	.2	[3] .1	.1	[4] .14	.5	0.5		0.2	.5		[3] 20,000			
SO_2	Conductivity	.2	[3] .1		[4] .14	.5		750							
SO_2	Spectrophotometric-gas phase.				[4] .14	.5	.5			.5	0.2				
O_3	Chemiluminescent			[3] .1				750		[4] .08		[3] 20,000			
O_3	Electrochemical		[3] .1		.5	.5				[4] .08		[3] 20,000			
O_3	Spectrophotometric-wet chemical (potassium iodide reaction).		[3] .1		.5	.5	[3] .5			[4] .08					
O_3	Spectrophotometric-gas phase.				.5	.5	[3] .5			[4] .08					
CO	Infrared							750				20,000	[4] 10		
CO	Gas chromatography with flame ionization detector.											20,000	[4] 10		0.5
CO	Electrochemical						.5		.2			20,000	[4] 10		
CO	Catalytic combustion-thermal detection.		.1					750	.2			20,000	[4] 10	5.0	.5
CO	IR fluorescence							750				20,000	[4] 10		.5
CO	Mercury replacement UV photometric.								.2				[4] 10		.5
NO_2	Chemiluminescent		[3] .1		.5	[4] .1	.5					20,000			
NO_2	Spectrophotometric-wet chemical (azo-dye reaction).				.5	[4] .1	.5	750		.5					
NO_2	Electrochemical	0.2	[3] .1		.5	[4] .1	.5	750		.5		20,000	50		
NO_2	Spectrophotometric-gas phase.		[3] .1		.5	[4] .1	.5			.5		20,000	50		

[1] Concentrations of interferant listed must be prepared and controlled to ±10 percent of the state value.
[2] Analyzer types not listed will be considered by the administrator as special cases.
[3] Do not mix with pollutant.
[4] Concentration of pollutant used for test. These pollutant concentrations must be prepared to ±10 percent of the stated value.

(A) Service or perform the indicated maintenance on the scrubber or prefilter as directed in the manual referred to in § 53.4(b)(3).

(B) Before testing for each interferent, allow the test analyzer to sample through the scrubber a test atmosphere containing the interferent at a concentration equal to the value specified in table B–3. Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to testing.

(iii) Generate three test atmosphere streams as follows:

(A) Test atmosphere *P:* Pollutant concentration.

(B) Test atmosphere *I:* Interference concentration.

(C) Test atmosphere *Z:* Zero air.

(iv) Adjust the individual flow rates and the pollutant or interferent generators for the three test atmospheres as follows:

(A) The flow rates of test atmospheres *I* and *Z* shall be identical.

(B) The concentration of pollutant in test atmosphere *P* shall be adjusted such that when *P* is mixed (diluted) with either test atmosphere *I* or *Z*, the resulting concentration of pollutant shall be as specified in table B–3.

(C) The concentration of interferent in test atmosphere *I* shall be adjusted such that when *I* is mixed (diluted) with test atmosphere *P*, the resulting concentration of interferent shall be equal to the value specified in table B–3.

(D) To minimize concentration errors due to flow rate differences between *I* and *Z*, it is recommended that, when possible, the flow rate of *P* be from 10 to 20 times larger than the flow rates of *I* and *Z*.

(v) Mix test atmospheres *P* and *Z* by passing the total flow of both atmospheres through a mixing flask.

(vi) Sample and measure the mixture of test atmospheres *P* and *Z* with the test analyzer. Allow for a stable reading, and record the reading, in concentration units, as *R* (see Figure B–3).

(vii) Mix test atmospheres *P* and *I* by passing the total flow of both atmospheres through a mixing flask.

(viii) Sample and measure this mixture. Record the stable reading, in concentration units, as R_I.

(ix) Calculate the interference equivalent (*IE*) as:

$$IE = R_I - R$$

IE must be equal to or less than the specification given in table B–1 for each interferent to pass the test.

(x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each interferent.

(xi) For those interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B–3, adjust the concentration of test atmosphere *I* to the specified value without being mixed or diluted by the pollutant test atmosphere. Determine *IE* as follows:

(A) Sample and measure test atmosphere *Z* (zero air). Allow for a stable reading and record the reading, in concentration units, as *R*.

(B) Sample and measure the interferent test atmosphere *I*. If the test analyzer is not capable of negative readings, adjust the analyzer (not the recorder) to give an offset zero. Record the stable reading in concentration units as R_I, extrapolating the calibration curve, if necessary, to represent negative readings.

(C) Calculate $IE = R_I - R$. *IE* must be equal to or less than the specification in table B–1 to pass the test.

(xii) Sum the absolute value of all the individual interference equivalents. This sum must be equal to or less than the total interferent specification given in table B–1 to pass the test.

(e) *Zero drift, span drift, lag time, rise time, fall time, and precision*—(1) *Technical definitions*—(i) *Zero drift:* The change in response to zero pollutant concentration, over 12- and 24-hour periods of continuous unadjusted operation.

(ii) *Span drift:* The percent change in response to an up-scale pollutant concentration over a 24-hour period of continuous unadjusted operation.

(iii) *Lag time:* The time interval between a step change in input concentration and the first observable corresponding change in response.

(iv) *Rise time:* The time interval between initial response and 95 percent of final response after a step increase in input concentration.

(v) *Fall time:* The time interval between initial response and 95 percent of final response after a step decrease in input concentration.

(vi) *Precision:* Variation about the mean of repeated measurements of the same pollutant concentration, expressed as one standard deviation about the mean.

(2) Tests for these performance parameters shall be accomplished over a period of seven (7) or more days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be varied from day to day. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days as necessary. The tests are performed sequentially in a single procedure.

(3) The 24-hour test day may begin at any clock hour. The first 12 hours out of each test day are required for testing 12-hour zero drift. Tests for the other parameters shall be conducted during the remaining 12 hours.

(4) Table B–4 specifies the line voltage and room temperature to be used for each test day. The line voltage and temperature shall be changed to the specified values at the start of each test day (i.e., at the start of the 12-hour zero test). Initial adjustments (day zero) shall be made at a line voltage of 115 volts (rms) and a room temperature of 25 °C.

(5) The tests shall be conducted in blocks consisting of 3 test days each until 7 or 15 test results have been obtained. (The final block may contain fewer than three test days.) If a test is interrupted by an occurrence other than a malfunction of the test analyzer, only the block during which the interruption occurred shall be repeated.

(6) During each block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments which the test analyzer performs by itself are permitted at any time.

(7) At least 4 hours prior to the start of the first test day of each block, the test analyzer may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual referred to in § 53.4(b)(3). If a new block is to immediately follow a previous block, such adjustments or servicing may be done immediately after completion of the day's tests for the last day of the previous block and at the voltage and temperature specified for that day, but only on test days 3, 6, 9, and 12.

NOTE: If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as necessary to complete the service or adjustment operation.

(8) All response readings to be recorded shall first be converted to concentration units according to the calibration curve. Whenever a test atmosphere is to be measured but a stable reading is not required, the test atmosphere shall be measured long enough to cause a change in response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart. (See Figure B–1 illustrating the pattern of the required readings.)

TABLE B–4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS

Test day	Line voltage,[1] rms	Room temperature,[2] °C	Comments
0	115	25	Initial set-up and adjustments.
1	125	20	
2	105	20	
3	125	30	Adjustments and/or periodic maintenance permitted at end of tests.
4	105	30	
5	125	20	
6	105	20	Adjustments and/or periodic maintenance permitted at end of tests.
7	125	30	Examine test results to ascertain if further testing is required.
8	105	30	
9	125	20	Adjustments and/or periodic maintenance permitted at end of tests.
10	105	20	
11	125	30	
12	105	30	Adjustments and/or periodic maintenance permitted at end of tests.

TABLE B–4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS—Continued

Test day	Line voltage,[1] rms	Room temperature,[2] °C	Comments
13	125	20	
14	105	20	
15	125	30	

[1] Voltage specified shall be controlled to ±1 volt.
[2] Temperature specified shall be controlled to ±1 °C.

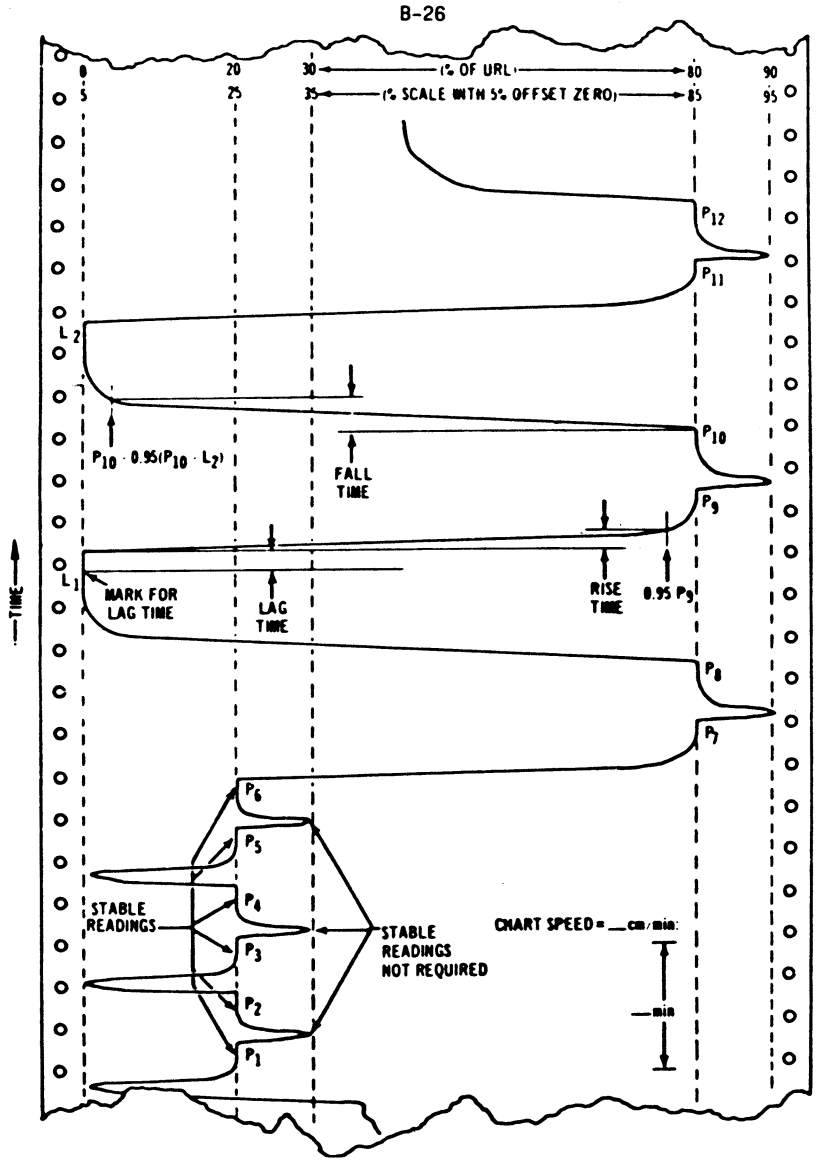

Figure B-1. Example showing the nature of the tracing obtained during the test for drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

(9) *Test procedure.* (i) Arrange to generate pollutant test atmospheres as follows:

Test atmosphere	Pollutant concentration (percent)
A_0	Zero air.
A_{20}	20±5 of the upper range limit.
A_{30}	30±5 of the upper range limit.

Test atmosphere	Pollutant concentration (percent)
A_{80}	80±5 of the upper range limit.
A_{90}	90±5 of the upper range limit.

Test atmospheres A_0, A_{20}, and A_{80} shall be consistent during the tests and from day to day.

(ii) For steps (xxv) through (xxxi) of this section, a chart speed of at least 10 centimeters per hour shall be used. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.

(iii) Allow sufficient time for test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Recalibrate, if necessary, and adjust the zero baseline to 5 percent of chart. No further adjustments shall be made to the analyzer until the end of the tests on the third day.

(iv) Measure test atmosphere A_0 until a stable reading is obtained, and record this reading (in ppm) as Z'_n, where n = 0 (see Figure B–4 in appendix A).

(v) Measure test atmosphere A_{20}. Allow for a stable reading and record it as M'_n, where n = 0.

(vi) Measure test atmosphere A_{80}. Allow for a stable reading and record it as S'_n, where n = 0.

(vii) The above readings for Z'_0, M'_0, and S'_0 should be taken at least four (4) hours prior to the beginning of test day 1.

(viii) At the beginning of each test day, adjust the line voltage and room temperature to the values given in table B–4.

(ix) Measure test atmosphere A_0 continuously for at least twelve (12) continuous hours during each test day.

(x) After the 12-hour zero drift test (step ix), sample test atmosphere A_0. A stable reading is not required.

(xi) Measure test atmosphere A_{20} and record the stable reading (in ppm) as P_1. (See Figure B–4 in appendix A.)

(xii) Sample test atmosphere A_{30}; a stable reading is not required.

(xiii) Measure test atmosphere A_{20} and record the stable reading as P_2.

(xiv) Sample test atmosphere A_0; a stable reading is not required.

(xv) Measure test atmosphere A_{20} and record the stable reading as P_3.

(xvi) Sample test atmosphere A_{30}; a stable reading is not required.

(xvii) Measure test atmosphere A_{20} and record the stable reading as P_4.

(xviii) Sample test atmosphere A_0; a stable reading is not required.

(xix) Measure test atmosphere A_{20} and record the stable reading as P_5.

(xx) Sample test atmosphere A_{30}; a stable reading is not required.

(xxi) Measure test atmosphere A_{20} and record the stable reading as P_6.

(xxii) Measure test atmosphere A_{30} and record the stable reading as P_7.

(xxiii) Sample test atmosphere A_{90}; a stable reading is not required.

(xxiv) Measure test atmosphere A_{80} and record the stable reading as P_8. Increase chart speed to at least 10 centimeters per hour.

(xxv) Measure test atmosphere A_0. Record the stable reading as L_1.

(xxvi) Quickly switch the test analyzer to measure test atmosphere A_{80} and mark the recorder chart to show the exact time when the switch occurred.

(xxvii) Measure test atmosphere A_{90} and record the stable reading as P_{80}.

(xxviii) Sample test atmosphere A_{90}; a stable reading is not required.

(xxix) Measure test atmosphere A_{80} and record the stable reading as P_{10}.

(xxx) Measure test atmosphere A_0 and record the stable reading as L_2.

(xxxi) Measure test atmosphere A_{80} and record the stable reading as P_{11}.

(xxxii) Sample test atmosphere A_{90}; a stable reading is not required.

(xxxiii) Measure test atmosphere A_{80} and record the stable reading as P_{12}.

(xxxiv) Repeat steps (viii) through (xxxiii) of this section, each test day.

(xxxv) If zero and span adjustments are made after the readings are taken on test days 3, 6, 9, or 12, complete all adjustments; then measure test atmospheres A_0, A_{80}, and A_{20}. Allow for a stable reading on each, and record the readings as $Z'_nS'_n$, and M_n respectively, where n = the test day number.

(10) Determine the results of each day's tests as follows. Mark the recorder chart to show readings and determinations.

(i) *Zero drift.* (A) 12-hour. Examine the strip chart pertaining to the 12-

hour continuous zero air test. Determine the minimum (Cmin.) and maximum (Cmax.) readings (in p/m) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Determine the 12-hour zero drift (12*ZD*) as $12ZD = C^{max.} - C^{min.}$. (See Figure B–5 in appendix A.)

(B) Calculate the 24-hour zero drift (24*ZD*) for the *n*-th test day as $24ZD_n = Z_n - Z_{n-1}$, or $24ZD_n = Z_n - Z'_{n-1}$ if zero adjustment was made on the previous day, where $Z_n = \frac{1}{2}(L_1+L_2)$ for L_1 and L_2 taken on the *n*-th test day.

(C) Compare 12*ZD* and 24*ZD* to the zero drift specification in table B–1. Both 12*ZD* and 24*ZD* must be equal to or less than the specified value to pass the test for zero drift.

(ii) *Span drift.* (A) Span drift at 20 percent of URL (MSD)

$$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$$

$$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$M_n = \frac{1}{6}\sum_{i=1}^{6} P_i$$

n indicates the *n*-th test day, and *i* indicates the *i*-th reading on the *n*-th day.

(B) Span drift at 80 percent of URL (USD):

$$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$$

or

$$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$S_n = \frac{1}{6}\sum_{i=7}^{12} P_i$$

n indicates the *n*-th test day, and *i* indicates the *i*-th reading on the *n*-th test day.

(C) Both USD and MSD must be equal to or less than the respective specifications given in table B–1 to pass the test for span draft.

(iii) *Lag time.* Determine, from the strip chart, the elapsed time in minutes between the mark made in step (xxvi) and the first observable (two times the noise level) response. This time must be equal to or less than the time specified in table B–1 to pass the test for lag time.

(iv) *Rise time.* Calculate 95 percent of reading P_9 and determine from the recorder chart, the elapsed time between the first observable (two times noise level) response and a response equal to 95 percent of the P_9 reading. This time must be equal to or less than the rise time specified in table B–1 to pass the test for rise time.

(v) *Fall time.* Calculate five percent of $(P_{10} - L_2)$ and determine, from the strip chart, the elapsed time in minutes between the first observable decrease in response following reading P_{10} and a response equal to five percent of $(P_{10} - L_2)$. This time must be equal to or less than the fall time specification in table B–1 to pass the test for fall time.

(vi) *Precision.* Calculate precision (P_{20} and P_{80}) for each day's test as follows:

(A)

$$P_{30} = \sqrt{\frac{1}{5}\left[\sum_{i=1}^{6} P_i^2 - \frac{1}{6}\left(\sum_{i=1}^{6} P_i\right)^2\right]}$$

(B)

$$P_{30} = \sqrt{\frac{1}{5}\left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6}\left(\sum_{i=7}^{12} P_i\right)^2\right]}$$

(C) Both P_{20} and P_{80} must be equal to or less than the specification given in table B–1 to pass the test for precision.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52694, Dec. 1, 1976]