

RELATIVE DENSITY OF GAS BY SCHILLING EFFUSION METHOD

UOP Method 114-86

SCOPE

This method is for determining the relative density of hydrogen and other gases whose relative densities do not exceed 1.3. The relative density of air is defined as unity.

OUTLINE OF METHOD

The respective effusion times for air and the sample to pass through the same orifice under identical conditions are measured. The relative density is calculated and reported on either a wet (uncorrected) or dry (corrected) basis.

APPARATUS

Barometer, aneroid, graduated in mm of Hg, Sargent-Welch Scientific, Cat. No. S-4631, or equivalent

Schilling effusimeter, Stanhope-Seta Ltd., Cat. No. 97300-0; consisting of (see Figure for assembly):

Gas tube, glass, calibrated, 50-mL

Jar, glass, 30.5- $\times$ 7.6-cm in diameter (typical dimensions)

Orifice plate, platinum or stainless steel, 0.05-0.1-mm thick, with an orifice of 0.10-0.15 mm in diameter

Stopcock, three-way

Stopwatch, 60 minutes, 0.2 second, Sargent-Welch Scientific, Cat. No. S-77446, or equivalent

Thermometer, total immersion, -35 to 50 C, Sargent-Welch Scientific, Cat. No. S-80005A, or equivalent

Tubing, rubber, 6.4-mm ID, 9.5-mm OD, Sargent-Welch Scientific, Cat. No S-73505D, or equivalent

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

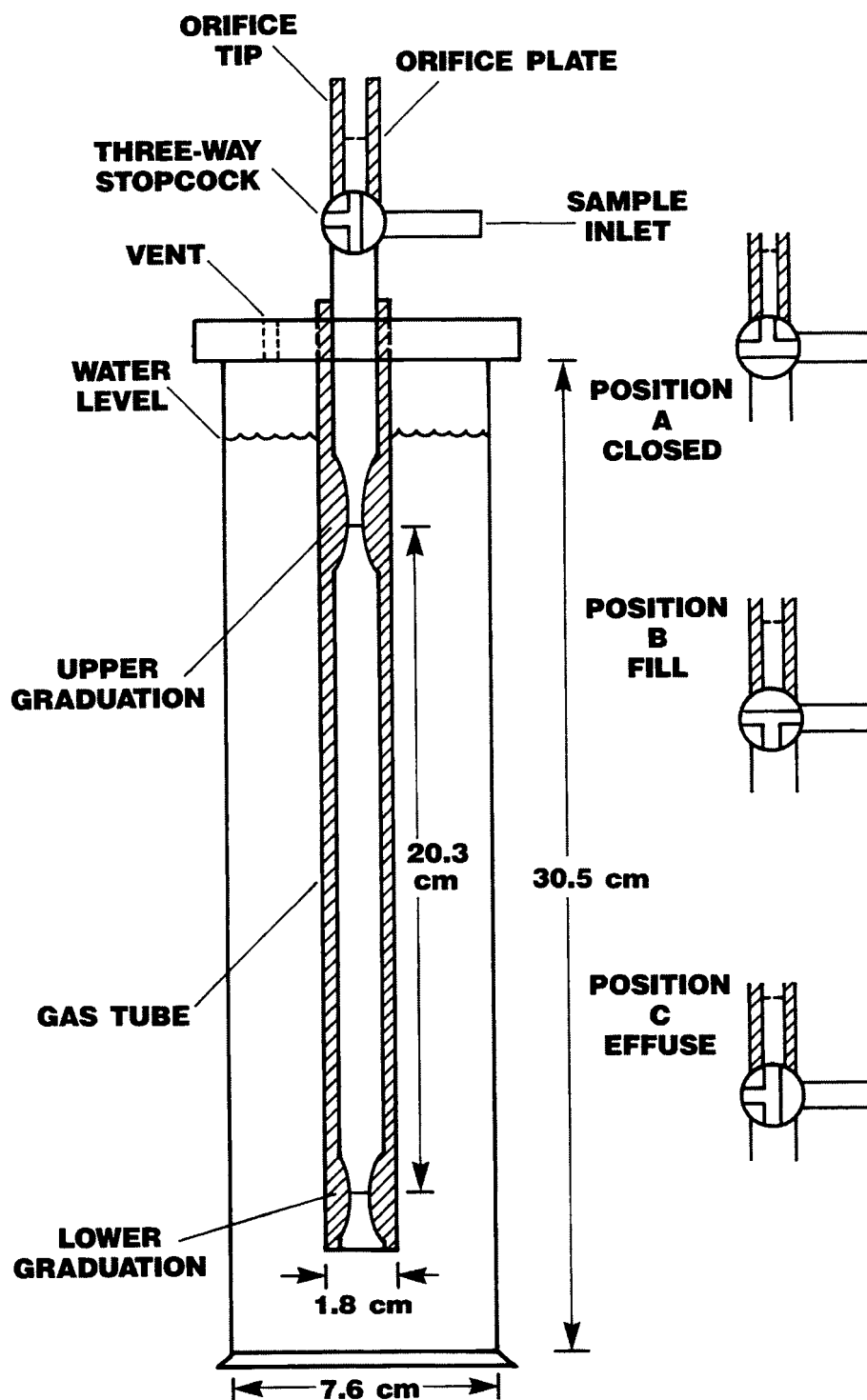


Figure
Schilling Effusimeter

PROCEDURE

Remove the cap from the orifice tip and screw the orifice tip to the top of the three-way stopcock on the glass tube. Remove the vent cap at the top of the glass jar and leave it off throughout the test procedure. Fill the jar with clear tap water to approximately 5 mm above the upper graduation on the glass tube. Record the temperature of the water and the atmospheric pressure. Set the stopcock in Position B, withdraw the gas tube from the jar and allow it to fill with air. Set the three-way stopcock to Position A and replace the tube in the jar. Wait one minute for the air and water temperature to equilibrate. Open the three-way stopcock (Position C) so that air will be expelled through the orifice and, using a stopwatch, measure the time required for the water to rise from the lower graduation to the upper graduation etched on the glass tube. Record the time to the nearest 0.2 second. Repeat the procedure five times. Test the system for leaks if check determinations cannot be obtained within 0.5 seconds. Average the effusion times of the five trials.

Connect a length of rubber tubing from the gas source to the stopcock inlet. Set the stopcock in Position B and allow the gas sample to pass downward through the glass tube. Allow the gas to bubble from the bottom of the tube through the water and vent to the atmosphere for 3 to 4 minutes through the vent hole. It is necessary to bubble gas through the system when changing from air to the sample, or from sample to sample, to saturate the water with the sample gas to ensure that traces of the previous sample do not produce erroneous readings. Replace the water if it becomes contaminated with particulate or other soluble materials. Control the gas flow so that no water is lost from the jar as the result of violent bubbling of the gas. Maintain a constant water level during air and gas effusion time determinations for the same test.

Close the stopcock (Position A) and shut off the gas. Allow the gas in the glass tube to equilibrate one minute. Open the three-way stopcock to the orifice (Position C), and record the time required for the water level to rise from the lower to the upper graduation. Repeat the measurement five times. Test the system for leaks if check determinations cannot be obtained within 0.5 seconds. Average the effusion times for the five trials.

CALCULATIONS

Calculate the uncorrected relative density of the gas according to the following equation (see *APPENDIX* for derivation):

$$S = \frac{G^2}{A^2}$$

where:

- A** = average effusion time of air, seconds
- G** = average effusion time of gas, seconds
- S** = relative density of gas, uncorrected

Calculate the relative density (dry basis) using the following equation:

$$S_d = \frac{S(P + H - 0.38P_w) - S_wP_w}{(P + H) - P_w}$$

where:

- H** = hydrostatic pressure, typically 15-mm
P = atmospheric pressure, mm
P_w = vapor pressure of water at T, mm (see Table)
T = test temperature, C
S = relative density of gas, uncorrected
S_d = relative density of gas, dry
S_w = relative density of water vapor at T, typically 0.622
0.38 = constant, 1.0 – 0.62 (relative density of water vapor at T)

NOTES

1. Keep the cap over the orifice when not in use. Clean the orifice when necessary with alcohol. Examine the orifice periodically with a microscope. Replace it when it is corroded or burred.
2. Relative density is often used to determine hydrogen purity of recycle streams under field conditions. A calibration curve is prepared correlating relative density with percent hydrogen as determined by UOP Method 539, a gas chromatographic technique.

PRECISION

Based on six replicate determinations, the estimated standard deviation for two gas density ranges is listed below. Duplicate analyses by the same operator should not differ by more than the allowable difference shown (95% probability).

<u>Gas</u>	<u>Relative Density</u>	<u>esd</u>	<u>Allowable Difference</u>
Hydrogen	0.0879	0.0010	0.0036
Ethylene	0.8598	0.0021	0.0076

TIME FOR ANALYSIS

The elapsed time and labor requirement for one analysis are identical, 1.0 hour.

REFERENCES

CRC Handbook of Chemistry and Physics, 65th Edition, CRC Press, Boca Raton, FL, 1984, p. D-193
 Gas Engineers Handbook, The Industrial Press, New York, NY, 1966, Section 6, p. 48
 UOP Method 539

SUGGESTED SUPPLIERS

Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
 Stanhope-Seta Ltd., London Street, Chertsey, Surrey, England, KT16 8AP

APPENDIX

The calculations are based on the kinetic theory of gases from which Graham's Law is derived. The rate of effusion, the speed with which a gas will pass through a small opening, is inversely proportional to the square root of its molecular weight.

$$\frac{dV}{dt} = \left(\frac{RT}{2\pi M} \right)^{1/2}$$

where:

M = molecular weight

R = gas constant

T = temperature

t = time

V = volume

2, π = constants

The volume of the tube remains constant. Therefore:

$$\left(\frac{RT}{2\pi M_a} \right)^{1/2} t_a = \left(\frac{RT}{2\pi M_g} \right)^{1/2} t_g$$

where:

M_a = average molecular weight of air

M_g = average molecular weight of gas

t_a = effusion time of air

t_g = effusion time of gas

2, π , R and T are as previously defined

Substituting relative density for molecular weight and simplifying:

$$S_g = \frac{t_g^2}{t_a^2} \times S_a$$

where:

S_a = relative density of air

S_g = relative density of gas

t_a and **t_g** are as previously defined

The relative density of air is defined as 1.

TABLE

Vapor Pressure of Water Below 100 C

Pressure of aqueous vapor over water in mm of Hg for temperatures from
 -15.8 to 100 C. Values for fractional degrees between 50 and 89 were
 obtained by interpolation.

Temp. C	0.0	0.2	0.4	0.6	0.8	Temp. C	0.0	0.2	0.4	0.6	0.8
-15	1.436	1.414	1.390	1.368	1.345	42	61.50	62.14	62.80	63.46	64.12
-14	1.560	1.534	1.511	1.485	1.460	43	64.80	64.58	66.16	66.86	67.56
-13	1.691	1.665	1.637	1.611	1.585	44	68.26	68.97	69.69	70.41	71.14
-12	1.834	1.804	1.776	1.748	1.720	45	71.88	72.62	73.36	74.12	74.88
-11	1.987	1.955	1.924	1.893	1.863	46	75.65	76.43	77.21	78.00	78.80
-10	2.149	2.116	2.084	2.050	2.018	47	79.60	80.41	81.23	82.05	82.87
-9	2.326	2.289	2.254	2.219	2.184	48	83.71	84.56	85.42	86.28	87.14
-8	2.514	2.475	2.437	2.399	2.362	49	88.02	88.90	89.79	90.69	91.59
-7	2.715	2.674	2.633	2.593	2.553	50	92.51	93.5	94.4	95.3	96.3
-6	2.931	2.887	2.843	2.800	2.757	51	97.20	98.2	99.1	100.1	101.1
-5	3.163	3.115	3.069	3.022	2.976	52	102.09	103.1	104.1	105.1	106.2
-4	3.410	3.359	3.309	3.259	3.211	53	107.20	108.2	109.3	110.4	111.4
-3	3.673	3.620	3.567	3.514	3.461	54	112.51	113.6	114.7	115.8	116.9
-2	3.956	3.898	3.841	3.785	3.730	55	118.04	119.1	120.3	121.5	122.6
-1	4.258	4.196	4.135	4.075	4.016	56	123.80	125.0	126.2	127.4	128.6
0	4.579	4.513	4.448	4.385	4.320	57	129.82	131.0	132.3	133.5	134.7
1	4.926	4.847	4.765	4.685	4.605	58	136.08	137.3	138.5	139.9	141.2
2	5.294	5.200	5.115	5.030	4.945	59	142.60	143.9	145.2	146.6	148.0
3	5.685	5.585	5.490	5.395	5.300	60	149.38	150.7	152.1	153.5	155.0
4	6.101	6.000	5.900	5.800	5.700	61	156.43	157.8	159.3	160.8	162.3
5	6.543	6.435	6.330	6.225	6.120	62	163.77	165.2	166.8	168.3	169.8
6	7.013	6.900	6.790	6.680	6.570	63	171.38	172.9	174.5	176.1	177.7
7	7.513	7.395	7.280	7.165	7.050	64	179.31	180.9	182.5	184.2	185.8
8	8.045	7.920	7.795	7.670	7.545	65	187.54	189.2	190.9	192.6	194.3
9	8.609	8.475	8.345	8.210	8.080	66	196.09	197.8	199.5	201.3	203.1
10	9.209	9.065	8.925	8.785	8.645	67	204.96	206.8	208.6	210.5	212.3
11	9.844	9.690	9.540	9.390	9.240	68	214.17	216.0	218.0	219.9	221.8
12	10.518	10.355	10.195	10.035	9.875	69	223.73	225.7	227.7	229.7	231.7
13	11.231	11.060	10.890	10.720	10.550	70	233.7	235.7	237.7	239.7	241.8
14	11.987	11.805	11.625	11.445	11.265	71	243.9	246.0	248.2	250.3	252.4
15	12.788	12.595	12.405	12.215	12.025	72	254.6	256.8	259.0	261.2	263.4
16	13.634	13.430	13.230	13.030	12.830	73	265.7	268.0	270.2	272.6	274.8
17	14.530	14.315	14.105	13.895	13.685	74	277.2	279.4	281.8	284.2	286.6
18	15.477	15.250	15.030	14.810	14.590	75	289.1	291.5	294.0	296.4	298.8
19	16.477	16.235	16.000	15.765	15.530	76	301.4	303.8	306.4	308.9	311.4
20	17.535	17.280	17.035	16.790	16.545	77	314.1	316.6	319.2	322.0	324.6
21	18.650	18.385	18.130	17.875	17.620	78	327.3	330.0	332.8	335.6	338.2
22	19.827	19.550	19.280	19.010	18.740	79	341.0	343.8	346.6	349.4	352.2
23	21.068	20.775	20.485	20.195	19.905	80	355.1	358.0	361.0	363.8	366.8
24	22.377	22.070	21.770	21.470	21.170	81	369.7	372.6	375.6	378.8	381.8
25	23.756	23.435	23.120	22.805	22.490	82	384.9	388.0	391.2	394.4	397.4
26	25.209	24.875	24.550	24.225	23.900	83	400.6	403.8	407.0	410.2	413.6
27	26.739	26.390	26.050	25.710	25.370	84	416.8	420.2	423.6	426.8	430.2
28	28.349	27.985	27.630	27.275	26.920	85	433.6	437.0	440.4	444.0	447.5
29	30.043	29.665	29.290	28.915	28.540	86	450.9	454.4	458.0	461.6	465.2
30	31.824	31.435	31.045	30.655	30.265	87	468.7	472.4	476.0	479.8	483.4
31	33.695	33.290	32.885	32.480	32.075	88	487.1	491.0	494.7	498.5	502.2
32	35.663	35.245	34.830	34.415	34.000	89	506.1	510.0	513.9	517.8	521.8
33	37.729	37.295	36.865	36.435	36.005	90	525.76	529.77	533.80	537.86	541.95
34	39.898	39.445	38.990	38.535	38.080	91	546.05	550.18	554.35	558.53	562.75
35	42.175	41.700	41.230	40.760	40.290	92	566.99	571.26	575.55	579.87	584.22
36	44.563	44.075	43.590	43.105	42.620	93	588.60	593.00	597.43	601.89	606.38
37	47.067	46.565	46.065	45.565	45.065	94	610.90	615.44	620.01	624.61	629.24
38	49.692	49.175	48.660	48.140	47.620	95	633.90	638.59	643.30	648.05	652.82
39	52.442	51.905	51.370	50.835	50.300	96	657.62	662.45	667.31	672.20	677.12
40	55.324	54.770	54.220	53.670	53.120	97	682.07	687.04	692.05	697.10	702.17
41	58.34	57.770	57.210	56.650	56.090	98	707.27	712.40	717.56	722.75	727.98
						99	733.24	738.53	743.85	749.20	754.58
						100	760.00	765.45	770.93	776.44	782.00
						101	787.57	793.18	798.82	804.50	810.21