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COMPOSITION CHANGES IN LIQUID OXYGEN AS A RESULT OF EVAPORATION AND RELATION TO FUEL-CELL OPERATION

by Robert W. Easter

Lewis Research Center
Cleveland, Ohio 44135

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16. Abstract

An expression is presented which may be used to estimate changes in composition as a consequence of evaporation during the storage of liquid oxygen. The sensitivity of fuel-cell systems to such changes is discussed, and sample calculations are presented.

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COMPOSITION CHANGES IN LIQUID OXYGEN AS A RESULT OF

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SUMMARY

Evaporation of liquid oxygen leads to changes in composition because of differences between the volatility of oxygen and the volatilities of impurities.

An expression is developed to enable prediction of such changes, which depend on the phase equilibrium behavior and on relative rates of liquid and vapor removal from the containing vessel.

Sample calculations are carried out for propellant grade liquid oxygen, and the likelihood and effect of composition changes in reference to fuel-cell system operation are discussed.

INTRODUCTION

Liquefied oxygen (LOX) is used for a variety of purposes both in aerospace and terrestrial applications. Since the source of the majority of available LOX is air, a number of atmospheric gases are commonly present as impurities.

Aerospace users generally procure LOX under three different specifications as to impurity levels or concentrations: the "fuel cell," "breathing," and "propellant" grades (ref. 1). Fuel-cell grade, as the name implies, has been used as the oxidant for Gemini and Apollo fuel-cell systems and is required to be at least 99.995 percent pure oxygen.

Some fuel-cell systems presently under development are, however, being designed to utilize propellant grade LOX as oxidant, because of cost and convenience considerations. Propellant grade LOX is required to be only 99.6 percent oxygen.

Breathing grade has the same requirement as propellant grade with respect to total impurities, but embodies more stringent requirements regarding certain impurities such as hydrocarbons, carbon monoxide, and nitrogen oxides.

Fuel-cell behavior is, in general, sensitive to impurities in both fuel and oxidant. However, most aerospace fuel-cell systems use cryogenically stored liquid hydrogen as fuel, and because of the very low temperature of liquid hydrogen the solubility of other materials is almost nil. Thus, cryogenically stored hydrogen tends to be inherently of high purity, and for that reason this work deals only with impurities in LOX.

Since impurity levels are two orders of magnitude higher in propellant grade LOX than in fuel-cell grade, intelligent design of advanced fuel-cell systems requires more detailed knowledge of LOX composition and possible changes therein than was previously required. In particular, it is known that the volatilities of impurities in LOX differ from the volatility of LOX itself, and thus composition changes will result from partial vaporization of stored propellant grade LOX. It is the goal of this work to derive a means of estimating such changes and to investigate the likelihood of such changes occurring and their possible effects on fuel-cell system operation.

DERIVATION OF EXPRESSION TO PREDICT COMPOSITION CHANGES

Consider the vessel of figure 1. Mass balances on the liquid and vapor phases yield

$$-w_{LG} - w_L = \frac{dn_L}{dt} \tag{1}$$

$$w_{LG} - w_G = \frac{dn_G}{dt} \tag{2}$$

where w_{LG} is the molar flow rate of material leaving the liquid phase and entering the gas phase, w_L is the molar flow rate of liquid leaving the vessel, w_G is the molar flow rate of gas leaving the vessel, and n_L and n_G are the moles of liquid and gas in the vessel.

If V_L and V_G are the liquid and gas volumes and v_L and v_G are the mean molar volumes for the liquid and gas, then

$$n_L = \frac{V_L}{v_L}$$

$$n_G = \frac{V_G}{v_G}$$

Also, if

$$V_L + V_G = V_{total} = \text{a constant}$$

and if v_L and v_G are assumed constant, then

$$\frac{dn_L}{dt} = \frac{1}{v_L}\frac{dV_L}{dt} = \frac{-1}{v_L}\frac{dV_G}{dt} = -\frac{v_G}{v_L}\frac{dn_G}{dt}$$

and equations (1) and (2) may be combined to yield

$$w_{LG} = \frac{w_G + w_L\dfrac{v_L}{v_G}}{1 - \dfrac{v_L}{v_G}} \tag{3}$$

From the conservation of species i in the liquid phase,

$$-w_{LG}Y_i - w_L X_i = \frac{d}{dt}(X_i n_L) \tag{4}$$

where X_i is the mole fraction of species i in the liquid phase and Y_i is the mole fraction of species i in the vapor leaving the liquid phase.

Now if it is assumed that the liquid phase is homogeneous, that is, well mixed, and that evaporation takes place slowly, then Y_i may be taken to be the vapor mole fraction in equilibrium with the liquid.

Combining equations (1), (3), and (4) yields

$$\left(\frac{w_G + w_L\dfrac{v_L}{v_G}}{1 - \dfrac{v_L}{v_G}}\right)(X_i - Y_i) = n_L\frac{dX_i}{dt} \tag{5}$$

The equilibrium relation between X_i and Y_i may be written

$$Y_i = K_i X_i$$

where K_i is the distribution coefficient, which in general will be a function of temperature, pressure, and the liquid composition.

It is convenient to introduce a new independent variable ξ, which is the fractional amount of liquid remaining; that is,

$$\xi = \frac{n_L(t)}{n_L(0)}$$

Equation (5) may be written in terms of ξ, and the equilibrium relation embodied to yield

$$d \ln X_i = (K_i - 1) \left(\frac{w_G + w_L \dfrac{v_L}{v_G}}{w_G + w_L} \right) d \ln \xi \tag{6}$$

Equation (6) may be integrated with the condition

$$X_i = X_i(0) \quad \text{when} \quad \xi = 1$$

to give the composition variation in the liquid.

Rigorous application of equation (6) requires that K_i, w_G, and w_L be expressed in terms of ξ or X_i.

For most purposes, however, it is sufficient to break up the period of time over which the vessel is emptied into intervals over which the quantity $(K_i - 1)[w_G + w_L(v_L/v_G)/(w_G + w_L)]$ may be assumed constant. Thus, over the inverval I_j, where

$$I_j = (t_{j-1}, t_j)$$

w_G, w_L, K_i, and v_L/v_G are constant and equation (6) may be integrated to give

$$X_i(I_j) = X_i(t_{j-1})[\xi(I_j)]^{\theta(I_j)} \tag{7}$$

where $X_i(I_j)$ is the mole fraction of component i in the liquid during the jth interval, $X_i(t_{j-1})$ is the mole fraction at the end of the previous interval, $\xi(I_j)$ is given by

$$\xi(I_j) = \frac{n_L(t)}{n_L(t_{j-1})}$$

and $\theta(I_j)$ is given by

$$\theta = [K_i(I_j) - 1] \frac{1 + \dfrac{v_L}{v_G} \dfrac{w_L(I_j)}{w_G(I_j)}}{\dfrac{w_L(I_j)}{w_G(I_j)}}$$

It should be mentioned that equations (6) and (7) apply only if the liquid is unsaturated with respect to component i. If the liquid becomes saturated, X_i will no longer increase; further concentration of species i will result in the precipitation of a separate phase.

Equation (7) constitutes a simple relation which can be used to estimate composition changes in the contents of a vessel from which liquid and/or vapor is being withdrawn. After the necessary equilibrium data are presented, this relation will be applied to a particular situation, storage of propellant grade LOX.

At this point, however, it is well to mention the limitations on the calculational approach presented.

Equation (7) applies only to situations where LOX is in the subcritical state. LOX in the supercritical state will not undergo composition changes predicted by equation (7) because there will be no separate liquid and vapor phases with attendant propensities toward phase equilibrium.

In the derivation it was assumed that the liquid was well-mixed and that equilibrium evaporation took place. Therefore, composition changes predicted by equation (7) are the maximum attainable and represent limiting behavior, although such changes are probably closely approached under conditions of sufficiently low withdrawal rates.

EQUILIBRIUM DATA FOR IMPURITIES IN PROPELLANT GRADE LIQUID OXYGEN

The liquid oxygen for which composition change calculations were made was considered to contain the impurities listed in table I. This composition is fairly typical of commercially available propellant grade LOX (ref. 2, p. 19) and of the propellant grade LOX delivered to the NASA John F. Kennedy Space Center (unpublished data from S. Churchwell).

Carbon Dioxide

At temperatures near the normal boiling point of LOX, the vapor pressure of carbon dioxide (CO_2) is so low that CO_2 concentrates in the liquid as though it had no vapor pressure (ref. 2, p. 271). Thus, the distribution coefficient K_{CO_2} may be taken as zero. Moreover, since the solubility of CO_2 in LOX is quite low (see table II), solid CO_2 may be present if sufficient LOX is evaporated.

Methane

Methane (CH_4) equilibrium was investigated by Hodges and Burch (ref. 3). It is their observation that for liquid mixtures of oxygen (O_2) and CH_4 the distribution constant is essentially independent of composition for CH_4 levels of less than 1100 ppm. The CH_4 equilibrium in propellant grade LOX may thus be assumed that of the binary solution, and the distribution coefficient may be taken directly from the data presented (table III).

Although CH_4 will concentrate in LOX during evaporation, CH_4 and O_2 are essentially miscible in the liquid for temperatures at and above the normal boiling point (ref. 2, p. 268). Thus, no separate phase will appear.

Krypton

Krypton (Kr) equilibrium was investigated by Burch (ref. 4). Here too it was found that the distribution constants were composition independent for Kr levels of 1100 ppm or less. Thus, the binary system equilibrium data should be applicable to propellant grade LOX. Burch's data are given in table IV.

Although solubility data are unavailable, the solubility exceeds 10^5 ppm at 93.6 K since Burch measured equilibrium data at that level.

Nitrogen and Argon

A number of authors (refs. 5 to 7) have presented equilibrium data and correlations for the ternary system O_2, argon (Ar), and nitrogen (N_2), since such data are necessary in the design of air liquefacation and separation plants. The most convenient work for present purposes is that of Latimer (ref. 5). His correlations are in terms of the relative volatilities α_{ij}, where

$$\alpha_{ij} = \frac{y_i|x_i}{y_i|x_j}$$

Latimer presented the following expressions which would appear to predict available experimental relative volatilities in the region of interest to within ±5 percent:

$$\log \alpha_{N_2-O_2} = -0.470 + \frac{1}{T(K)}\left[88.4 + 8.3(X_{O_2} - X_{N_2}) + 2X_{AR}\right]$$

$$\log \alpha_{Ar-O_2} = -0.132 + \frac{1}{T(K)}\left[23.1 + 7.5(X_{O_2} - X_{Ar}) + 1.2X_{N_2}\right]$$

Investigation of these expressions shows that, for $X_{O_2} \geq 0.99$, $\alpha_{N_2-O_2}$ and α_{Ar-O_2} are essentially independent of composition (less than 1 percent variation). The effect of temperature is shown in table V.

From the definition of relative volatility, for the ternary system

$$Y_i = \frac{X_i\alpha_{iK}}{X_i\alpha_{iK} + X_j\alpha_{jK} + X_K}$$

and hence

$$K_{N_2} = \frac{\alpha_{N_2-O_2}}{X_{N_2}\alpha_{N_2-O_2} + X_{Ar}\alpha_{Ar-O_2} + X_{O_2}}$$

$$K_{Ar} = \frac{\alpha_{Ar-O_2}}{X_{Ar}\alpha_{Ar-O_2} + X_{N_2}\alpha_{N_2-O_2} + X_{O_2}}$$

For $X_{O_2} \geq 0.99$ the denominator in these expressions ranges from 1.00 to 1.03 for T = 90 K (the range is less for higher temperatures). Therefore, little error is introduced by assuming K_{N_2} and K_{Ar} constant for $X_{O_2} \geq 0.99$. In particular, it may be assumed that

$$K_{N_2} = 1.01\alpha_{N_2-O_2}$$

$$K_{Ar} = 1.01\alpha_{Ar\text{-}O_2}$$

The temperature dependency of K_{N_2} and K_{Ar} when these expressions are used is shown in table VI.

Since N_2 and Ar are both more volatile than O_2, the liquid phase will become dilute in N_2 and Ar and hence no separate phase will form.

Data on the oxygen molar volume ratio v_L/v_G are given in table VII (ref. 8).

It remains to specify the pressure-temperature behavior in the vessel. In most situations vessel emptying is probably accomplished more nearly isobarically than isothermally. Thus, for calculating composition changes, the pressure may be considered to remain constant. If the pressure is fixed, the equilibrium temperature will vary with composition. But over the range 0 to 99.99 percent vaporization of propellant grade LOX at 1 atmosphere, the boiling point of the residual liquid varies only 1 K (ref. 2, p. 271). The corresponding changes in distribution constants are only a few percent and may be neglected.

In summary, available equilibrium data indicate that composition change calculations for propellant grade LOX may be made by employing distribution constants which remain constant as long as the mole fraction of O_2 remains equal to or greater than 0.99. The results presented in the next section will show this to be the case until more than 99 percent of the liquid has been removed by evaporation.

RESULTS OF CALCULATIONS FOR PROPELLANT GRADE LIQUID OXYGEN

The data tabulated in the preceding section were used in conjunction with equation (7) to calculate composition changes for the following cases: case 1, evaporation (gaseous withdrawal) only at a total pressure of 1 atmosphere; case 2, evaporation only at a total pressure of 3 atmospheres; and case 3, an arbitrarily chosen withdrawal profile combining liquid and gaseous withdrawal at a total pressure of 1 atmosphere.

Figure 2 shows the results for case 1. A similar figure is presented in reference 2 (p. 272). However, no information is given on the method of calculation. Analysis of the figure in reference 2 (p. 272) shows that the same type of expression as equation (7) was used but that the distribution constants apparently employed differ from those used in this work by considerable amounts. It is presumed that the values used in this work are more accurate, being derived from more recent data.

The results for case 2 are given in figure 3. Figure 4 shows the withdrawal profile chosen to illustrate the effects of combined liquid and gaseous withdrawal. Note that the composition change calculations do not require absolute withdrawal rate information;

rather only the ratio of liquid withdrawal rate to gaseous withdrawal rate as a function of the fraction of liquid remaining is required.

Table VIII gives the form of equation (7) used in the calculations for each of the four intervals of case 3, and figure 5 shows the calculated results.

The results of cases 1 to 3 are presented in figures 2, 3, and 5 in terms of parts per million, defined as moles of species i times 10^6 divided by total moles. If solid CO_2 is present, it is considered to be dispersed in the liquid, and "total moles" in the definition is considered to be moles of liquid plus moles of solid.

It is apparent from figure 5 that significant composition changes occur only when the liquid is depleted by gaseous withdrawal. This is a consequence of the assumption of a well-mixed liquid; evaporation is the only mechanism by which composition changes may take place. When the liquid is depleted only by liquid withdrawal, just enough evaporation takes place to maintain the total pressure constant, and the associated composition changes are negligible (see expression for interval 2 in table VIII).

In cases where depletion is by evaporation only (figs. 2 and 3 and interval 4 of fig. 5), it follows from the definition of the distribution constant K_i that the impurities whose concentrations change the least are those whose distribution constants are close to unity. Impurities with distribution constants much greater than unity will disappear quickly from the liquid phase; those with distribution constants much less than unity will concentrate quickly.

Typical propellant grade LOX will become purer overall as evaporation proceeds, until somewhat more than 95 percent has evaporated, at which time the purity will begin to decrease rapidly because of the continued buildup of the nonvolatile impurities.

Finally, comparison of figures 2 and 3 shows that as the total pressure increases the changes in levels of impurities become somewhat less pronounced. Reference to tables III and IV reveals that this trend is due to the general tendency of the distribution constants to converge towards 1.0 with increasing temperature or pressure.

IMPLICATIONS WITH REGARD TO FUEL-CELL SYSTEM OPERATION

As pointed out in the section DERIVATION OF EXPRESSION TO PREDICT COMPOSITION CHANGES, equation (7) gives the maximum possible composition changes, which are most closely attained under conditions of low rates of withdrawal. In particular, the results of figure 2 should be quite representative of the situation where a tank of LOX is stored with no depletion other than venting of evaporation caused by the inherent heat-leak characteristics of the tank. If the LOX is stored for a length of time long enough that the amount remaining is considerably less than the initial amount, the concentrations of the impurities will differ considerably from the original values. If the

remaining LOX is subsequently used in an application which is sensitive to impurities, these changes may be important.

Such an application would be use as the oxidant in a fuel-cell system. From the point of view of effect on fuel-cell behavior, impurities in oxygen may be classed as either "active" or "inert." Active impurities are those which react chemically with some other component of the fuel cell, to the detriment of electrical performance, and inert impurities are those which may accumulate within the fuel cell, physically preventing oxygen from reaching sites at which it may be reduced electrochemically. Argon, nitrogen, and krypton behave as inerts in all fuel-cell systems; while in one important class of fuel cells, those with alkaline electrolytes, CO_2 and, provisionally CH_4, are considered active impurities (Ch_4 may, under some conditions, be converted to CO_2 within the fuel cell). The deleterious effects of CO_2 are described in reference 9, and the buildup of inerts in fuel-cell electrodes is treated in reference 10.

If, for instance, LOX is stored at 1 atmosphere until 70 percent of the original amount has evaporated, the level of inerts will have decreased by about 55 percent, but the level of active impurities (CO_2, solid CO_2, and CH_4) will have increased about 250 percent (fig. 2).

Thus, lengthy prestorage of LOX to be used eventually in a system designed around certain impurity levels should be avoided, not only because of the obvious waste involved, but also because of the off-design operation imposed by concentration changes.

It should be noted that the length of time required to evaporate, say, 70 percent of the LOX in a storage vessel depends on the heat-leak rate. The time required may thus vary from a few days for small laboratory Dewar-type flasks to perhaps years for large fixed storage tanks employing advanced cryogenic insulating techniques.

Concentration changes may also take place in LOX tankage from which O_2 is being drawn for use as fuel-cell oxidant. In general, the larger the portion removed by evaporation and gaseous withdrawal, the greater the changes in impurity levels. Table IX lists possible storage modes for LOX fuel-cell oxidant and ranks qualitatively the various modes in order of degree of composition change. The rankings merely reflect the degree to which the LOX is removed by evaporation.

For cases in which removal is by means of the gas phase, the gaseous impurity levels as a function of the fraction of liquid remaining ξ may be estimated from the equilibrium relation

$$Y_i = K_i X_i$$

This relation is applied to the results of figures 2 and 3 to yield figure 6, in which the levels of inerts and CH_4 in the gas phase are shown for 1- and 3-atmosphere storage pressures.

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Since the LOX storage mode is likely to be determined by considerations other than impurity concentration changes, fuel-cell system design should be flexible enough to handle changing impurity levels in the oxidant. By way of example, if inerts are removed from the fuel cell by periodic purging, a somewhat higher oxidant efficiency can be gained by making purge duration and/or frequency proportional to inert level, as opposed to making duration and/or frequency constant. For instance, if the gaseous O_2 composition varies as shown in figure 6, inerts will accumulate in the fuel cell at an ever decreasing rate, and a constant purge frequency and/or duration may not be sufficient to remove all inerts during the early stages of the mission and may be greater than necessary during the later stages of the mission.

On the other hand, figure 6 shows that the amount of CH_4 in the oxygen stream to the cell will increase rapidly in the later stages of LOX withdrawal. Thus, any in-line scrubbing apparatus (e.g., catalytic oxidizer to convert CH_4 to CO_2 and CO_2 sorber) should be designed to cope with the entire range of inlet CH_4 concentrations indicated by figure 6.

CONCLUSIONS

The following conclusions were drawn from a study of composition changes in liquid oxygen (LOX) as a result of evaporation and their relation to fuel-cell operation:

1. The levels of various impurities in LOX will change during subcritical storage if portions of the liquid are allowed to evaporate.

2. Composition changes during such storage and use may be estimated by a simple expression incorporating phase equilibrium data and the relative rates of withdrawal of liquid and vapor.

3. The degree to which LOX is removed from subcritical storage by the gas phase determines the amount of departure from the initially loaded composition. Thus, composition changes are greatest when all removal is by the gas phase and least when all removal is by the liquid phase.

4. Optimum operation of fuel-cell systems requires that impurity levels in reactants be accurately known. Reactants less pure than designed for will lead to performance degradation, while operation on purer reactants than designed for may impose unnecessary penalties in the form of excessive scrubbing capabilities and/or excessive purging. Therefore, the mode of LOX storage to be used should be taken into consideration in the design of fuel-cell systems.

Lewis Research Center,
    National Aeronautics and Space Administration,
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TABLE I. - INITIAL LIQUID-OXYGEN

IMPURITY LEVELS USED IN

SAMPLE CALCULATIONS

Impurity	Initial concentration	
	ppm	Mole fraction
CO_2	2	2×10^{-6}
CH_4	10	10^{-5}
Kr	10	10^{-5}
N_2	200	2×10^{-4}
Ar	2000	2×10^{-3}

TABLE II. - SOLUBILITY OF CARBON

DIOXIDE IN LIQUID OXYGEN

[Data from ref. 2, p. 20.]

Temperature, K	CO_2 solubility, ppm
90	4.4
95	5.1
100	6.0
105	7.0

TABLE III. - DISTRIBUTION CONSTANTS

FOR METHANE-OXYGEN MIXTURES

[Data from ref. 3.]

Temperature, K	Distribution constant, K_{CH_4}
107.15	0.352
100.15	.324
93.15	.299
90.0	[a].29

[a]Extrapolated.

TABLE IV. - DISTRIBUTION CONSTANTS

FOR KRYPTON-OXYGEN MIXTURES

[Data from ref. 4.]

Temperature, K	Distribution constant, K_{Kr}
106.5	0.168
101.35	.148
93.6	.122
90.0	[a].11

[a]Extrapolated.

TABLE V. - RELATIVE VOLATILITIES FOR

NITROGEN AND ARGON IN OXYGEN

[Mole fraction of oxygen $X_{O_2} \geq 0.99.$]

Temperature, K	Relative volatility	
	$\alpha_{N_2\text{-}O_2}$	$\alpha_{Ar\text{-}O_2}$
90	4.03	1.61
95	3.53	1.55
100	3.13	1.49
105	2.82	1.44

TABLE VI. - DISTRIBUTION CONSTANTS

FOR ARGON AND NIGROGEN

[Mole fraction of oxygen $X_{O_2} \geq 0.99.$]

Temperature, K	Distribution constant	
	K_{N_2}	K_{Ar}
90	4.07	1.63
95	3.57	1.57
100	3.16	1.51
105	2.85	1.45

TABLE VII. - OXYGEN MOLAR VOLUME

RATIO AT SATURATION

Pressure, atm	Temperature, K	Molar volume ratio, v_L/v_G
1	90.2	0.00392
2	97.4	.00766
3	102.2	.0115
4	105.9	.0153

TABLE VIII. - EXPRESSIONS USED IN

CASE 3 CALCULATIONS

Interval	Form of equation (7)
1	$X_i = X_i(\xi = 0) \cdot \xi^{(0.170)(K_i-1)}$
2	$X_i = X_i(\xi = 0.8)(1.25\ \xi)^{(0.00392)(K_i-1)}$
3	$X_i = X_i(\xi = 0.6)(1.67\ \xi)^{(0.0945)(K_i-1)}$
4	$X_i = X_i(\xi = 0.4)(2.50\ \xi)^{(K_i-1)}$

TABLE IX. - LIQUID-OXYGEN STORAGE MODES

State	Amount slated for fuel cell	Fuel-cell takeoff	Other takeoff	Degree of composition change (a)
Subcritical	Small fraction[b]	Liquid	Liquid	4
		Vapor	Liquid	3
Subcritical	Roughly one-half[c]	Liquid	Liquid	4
		Liquid	Vapor	2
		Vapor	Liquid	2
		Vapor	Vapor	1
Subcritical	All	Liquid	-----	4
		Vapor	-----	1
Supercritical	----------------	-----	-----	5

[a]Greatest change, 1; next greatest change, 2; etc.
[b]For example, propellant tankage.
[c]For example, combined fuel-cell and life-support tankage.
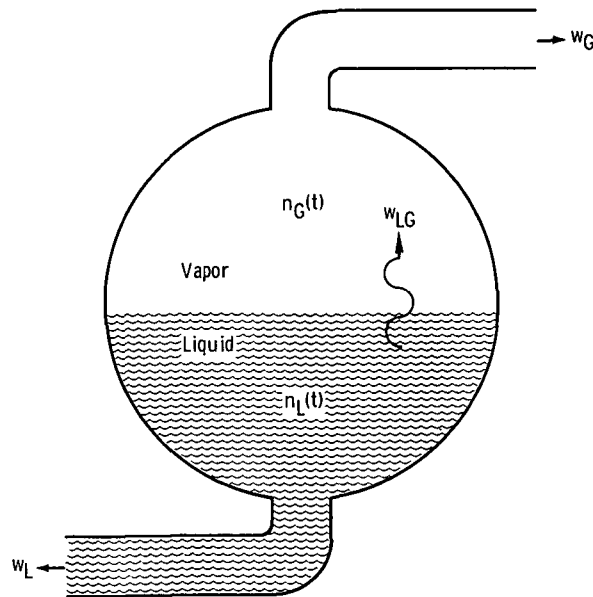
Figure 1. - Schematic representation of liquid-oxygen storage vessel.
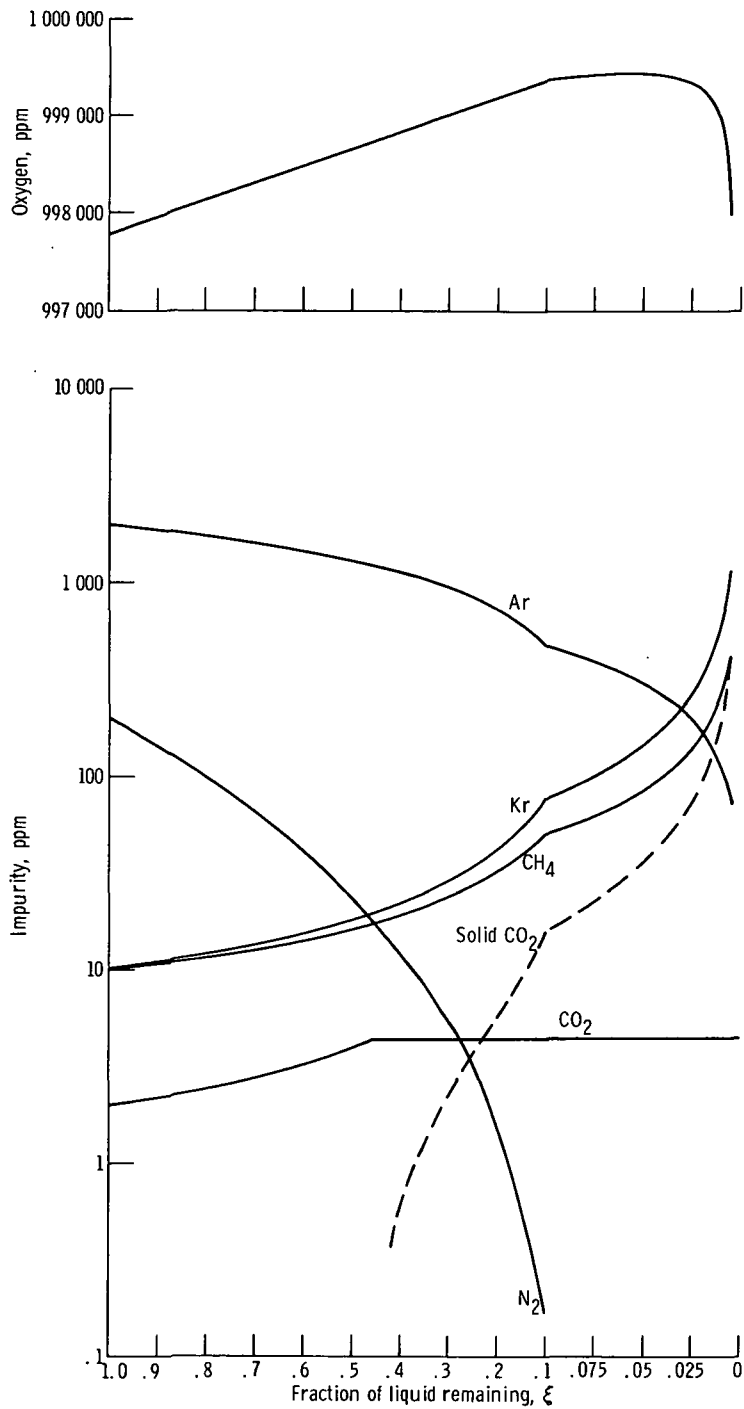
Figure 2. - Concentration changes in propellant-grade liquid oxygen as consequence of evaporation. No liquid removal; total pressure, 1 atmosphere.
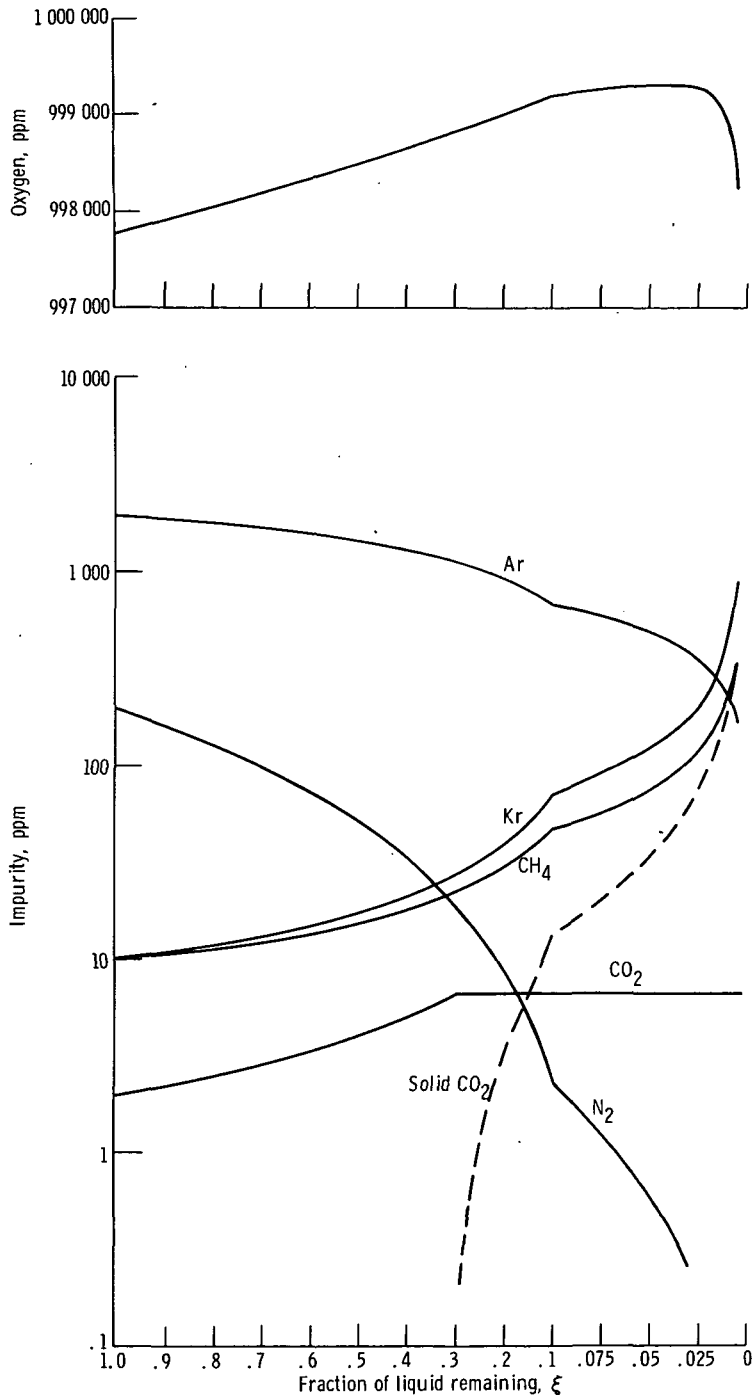
Figure 3. – Concentration changes in propellant-grade liquid oxygen as consequence of evaporation. No liquid removal; total pressure, 3 atmospheres.
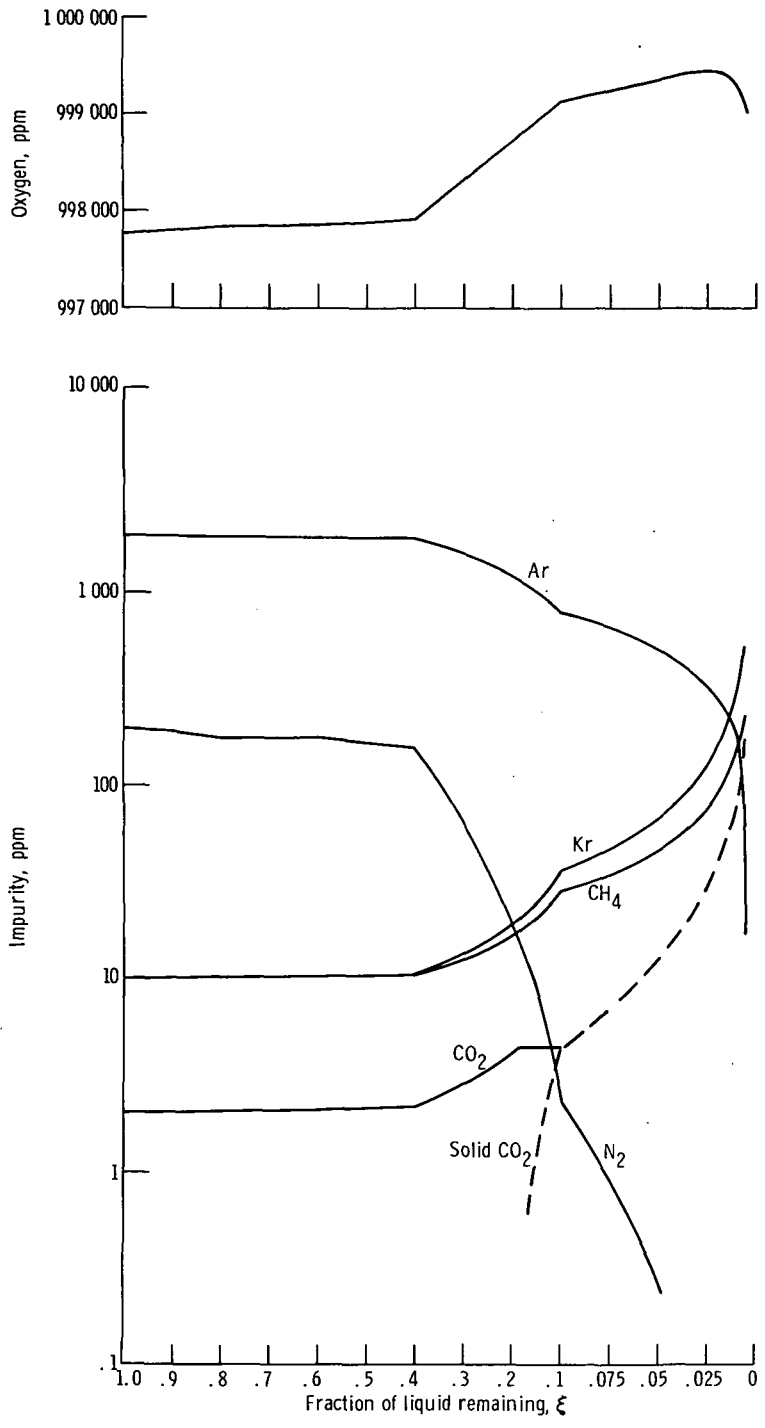
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Figure 5. - Concentration changes in propellant-grade liquid oxygen as consequence of evaporation. Arbitrary withdrawal profile; total pressure, 1 atmosphere.
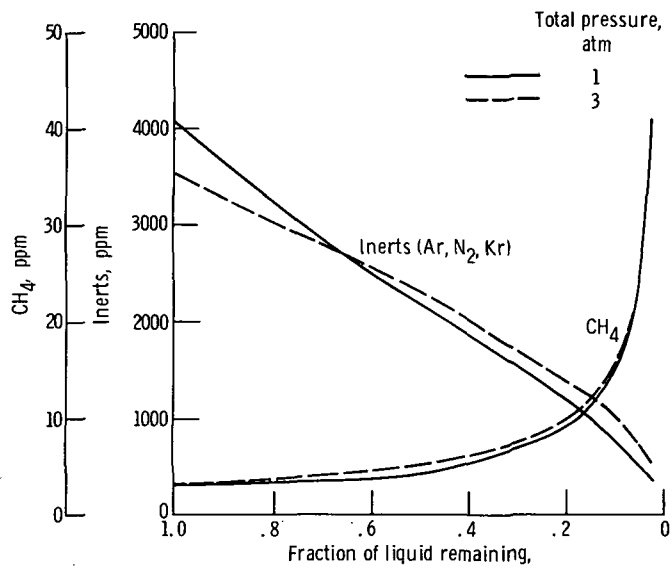
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Figure 6. - Propellant-grade liquid-oxygen boiloff composition changes (cases 1 and 2).

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