Registry No. 1-Butanol, 71-36-3; 2-methyl-1-propanol, 78-83-1; 2-butanol, 78-92-2; 2-methyl-2-propanol, 75-65-0; *n*-octane, 111-65-9; carbon tetrachloride, 56-23-5.

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Isopiestic Measurements of the Osmotic and Activity Coefficients for the System $HClO_4$–$Ba(ClO_4)_2$–H_2O at 25 °C

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Isopiestic measurements of the osmotic coefficients are presented for the system $HClO_4$–$Ba(ClO_4)_2$–H_2O. Parameters obtained from these results are used to calculate values of the osmotic coefficient and the activity coefficient of each component.

This paper is the third in a series on the Gibbs energies of mixed perchlorate solutions. The first paper (*1*) reported the results of isopiestic measurements on mixtures of univalent perchlorates. The second paper (*2*) reported the results of isopiestic measurements on mixtures of $HClO_4$ and $NaClO_4$ with $UO_2(ClO_4)_2$. This paper presents the results of such measurements on mixtures of $HClO_4$ and $Ba(ClO_4)_2$. The osmotic coefficient of $HClO_4$ increases strongly with ionic strength ($\phi = 4.39$ at $I = 15$) while that of $Ba(ClO_4)_2$ increases only slightly ($\phi = 1.79$ at $I = 15$). The mixture of these two should provide a good test of how well the osmotic coefficients of mixtures can be correlated.

Experimental Section

The experimental work described in this paper was performed in 1970. The delay in the analysis and presentation of the results was caused by a change in work assignment for the author.

Materials. The stock solution of H_2SO_4 was the same as used previously (*1*, *2*). The concentration was monitored by measuring the density to 5 parts in 100 000. The maximum change in molality from values used previously as indicated by these measurements was 0.06% over the course of these experiments.

The stock solution of NaCl was prepared from reagent grade crystals dried overnight at 200 °C. The concentration was calculated from the weight of the dried salt. Analysis by measurement of the density to 5 parts in 100 000 and comparison with the data of Wirth et al. (*3*) gave a difference in molality of 0.09%.

The stock solution of $HClO_4$ was prepared from reagent grade concentrated acid. The solution was analyzed by weight titration against Na_2CO_3. Analysis by measurement of the density to 5 parts in 100 000 and comparison with the data of Haase and Duecker (*4*) gave a molality 0.07% higher than the value obtained from the direct titration with Na_2CO_3.

The stock solution of $Ba(ClO_4)_2$ was prepared from reagent grade crystals. Flame photometric analysis of the solid crystals for strontium gave a value of 0.126% as Sr. Corrections were made for this impurity in all calculations. The concentration of the stock solution was determined by precipitation and weighing as $BaSO_4$ and determination of acid displaced from a H-form cation exchange resin. The molalities determined by these methods differed by 0.10%. A redetermination 2.5 years later by the $BaSO_4$ precipitation method gave a molality that was 0.23% higher than the earlier determination by this method. The concentration used was an average of the earlier and later measurements. Analysis by measurement of the density to 5 parts in 100 000 and comparison with the data of Kohner (*5*) and Willard and Smith (*6*) gave molalities that agreed by 0.01% for the earlier analyses and 0.08% for the later analyses.

Solutions of the various perchlorate mixtures were prepared by weight from the above solutions. Distilled water, passed through a mixed-bed ion-exchange column, was used for the preparation of all solutions.

Apparatus and Procedure. The apparatus and procedure have been described previously (*7*). The temperature of the bath varied 0.02 °C or less during the course of an equilibration. The temperature of the bath at equilibrium varied from one equilibration run to another within the range of 24.99–25.01 °C.

Following the weighing of the 12 dishes, the first dish was weighed again; the difference between the two weighings was less than 0.01% of the weight of the solution.

For each set of equilibrations there were at least two dishes containing the same solution. The maximum difference in concentration among the replicate dishes was 0.09%.

The reference material was NaCl up to the limit of its solubility and H_2SO_4 for higher concentrations. The osmotic coefficients for these electrolytes were taken from the tables given by Robinson and Stokes (*8*). The values used for the osmotic coefficient of NaCl differ from those derived from the results of Gibbard et al. (*9*) with an average deviation without regard to sign of 0.0012 and a maximum deviation of 0.0021. The values used for the osmotic coefficient of H_2SO_4 differ from those derived from the report of Staples (*10*) with an average deviation without regard to sign of 0.0045 and a maximum deviation of 0.0077.

Results

The concentrations at isopiestic equilibrium and the observed osmotic coefficients are given in Tables I–V. Concentrations are given as molality, *m*, and ionic strength on the molality

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Table I. Isopiestic Concentrations and Osmotic Coefficients for $Ba(ClO_4)_2$ with NaCl as Reference Material

	$Ba(ClO_4)_2$	
m_{ref}, mol/kg	m, mol/kg	ϕ_{obsd}
4.9408	2.7939	1.3993
4.8791	2.7581	1.3941
4.3086	2.4615	1.3289
4.2605	2.4331	1.3251
3.1778	1.8659	1.2002
3.0952	1.8247	1.1891
2.2088	1.3489	1.0866
2.1086	1.2961	1.0732
1.4716	0.9387	0.9981
1.3755	0.8846	0.9852
0.9848	0.6525	0.9406
0.9398	0.6263	0.9337
0.6688	0.4560	0.9043
0.6310	0.4326	0.8984

Table II. Isopiestic Concentrations and Osmotic Coefficients for $Ba(ClO_4)_2$ with H_2SO_4 as Reference Material

	$Ba(ClO_4)_2$	
m_{ref}, mol/kg	m, mol/kg	ϕ_{obsd}
7.4910	6.3260	1.9361
7.3406	6.1680	1.9248
7.3146	6.1487	1.9203
7.1485	5.9803	1.9056
6.4418	5.3160	1.8229
6.0946	4.9857	1.7820
6.0602	4.9490	1.7795
6.0168	4.9073	1.7745
5.2655	4.2280	1.6717
4.8596	3.8590	1.6135
4.6265	3.6580	1.5751
4.4390	3.4919	1.5470
4.1682	3.2633	1.5020
3.8514	2.9895	1.4514
3.4156	2.6289	1.3734
3.1805	2.4353	1.3312
3.1322	2.3988	1.3210
2.8758	2.1885	1.2771
2.3958	1.8098	1.1929
2.3468	1.7727	1.1836
2.1838	1.6441	1.1573
2.1234	1.5963	1.1478
1.6856	1.2620	1.0735
1.4974	1.1196	1.0428
1.2040	0.8992	0.9965
1.0326	0.7715	0.9698

Table III. Isopiestic Concentrations and Osmotic Coefficients for $HClO_4$ with H_2SO_4 as Reference Material

	$HClO_4$	
m_{ref}, mol/kg	m, mol/kg	ϕ_{obsd}
7.3406	7.2860	2.4441
7.1485	7.1155	2.4024
6.0602	6.1495	2.1481
6.0168	6.1050	2.1396
4.8596	5.0010	1.8676
4.4390	4.5875	1.7663
3.8514	3.9892	1.6315
3.1805	3.2995	1.4738
2.8758	2.9788	1.4074
2.1838	2.2568	1.2647
2.1234	2.1912	1.2543
1.4974	1.5405	1.1368
1.0326	1.0594	1.0594

Table IV. Isopiestic Concentrations and Osmotic Coefficients for the System $HClO_4$ (A)-$Ba(ClO_4)_2$ (B)-H_2O with NaCl as Reference Material

m_{ref}, mol/kg	I, mol/kg	y_B	ϕ_{obsd}
4.9408	4.3433	0.2499	1.5429
	5.1612	0.4999	1.5148
	6.3697	0.7497	1.4727
4.3086	3.8534	0.2499	1.4551
	4.5713	0.4999	1.4310
	5.6284	0.7497	1.3945
3.0952	2.8836	0.2499	1.2898
	3.4120	0.4999	1.2717
	4.1885	0.7497	1.2430
2.1086	2.0554	0.2499	1.1600
	2.4306	0.4999	1.1445
	2.9802	0.7497	1.1199
1.3755	1.4023	0.2499	1.0653
	1.6592	0.4999	1.0504
	2.0351	0.7497	1.0276
0.9398	0.9907	0.2499	1.0118
	1.1735	0.4999	0.9967
	1.4406	0.7497	0.9741
0.6310	0.6827	0.2499	0.9759
	0.8095	0.4999	0.9602
	0.9951	0.7497	0.9371

Table V. Isopiestic Concentrations and Osmotic Coefficients for the System $HClO_4$ (A)-$Ba(ClO_4)_2$ (B)-H_2O with H_2SO_4 as Reference Material

m_{ref}, mol/kg	I, mol/kg	y_B	ϕ_{obsd}
7.3406	8.6038	0.2499	2.3652
	10.486	0.4999	2.2642[a]
	13.397	0.7497	2.1263[a]
7.1485	8.3999	0.2499	2.3256
	10.215	0.4999	2.2310[a]
	13.024	0.7497	2.0996[a]
6.0602	7.2062	0.2499	2.0948
	8.6954	0.4999	2.0255
	10.959	0.7497	1.9282[a]
6.0168	7.1582	0.2499	2.0853
	8.6301	0.4999	2.0179
	10.871	0.7497	1.9220[a]
4.8596	5.8269	0.2499	1.8317
	6.9682	0.4999	1.7870
	8.6814	0.7497	1.7210
4.4390	5.3296	0.2499	1.7374
	6.3589	0.4999	1.6989
	7.8952	0.7497	1.6417
3.8514	4.6259	0.2499	1.6078
	5.4993	0.4999	1.5779
	6.7972	0.7497	1.5317
3.1805	3.8136	0.2499	1.4572
	4.5212	0.4999	1.4340
	5.5662	0.7497	1.3975
2.8758	3.4428	0.2499	1.3916
	4.0760	0.4999	1.3713
	5.0107	0.7497	1.3384
2.1838	2.6028	0.2499	1.2531
	3.0771	0.4999	1.2366
	3.7754	0.7497	1.2093
2.1234	2.5290	0.2499	1.2419
	2.9895	0.4999	1.2257
	3.6658	0.7497	1.1993
1.4974	1.7761	0.2499	1.1268
	2.0989	0.4999	1.1124
	2.5740	0.7497	1.0884
1.0326	1.2224	0.2499	1.0492
	1.4456	0.4999	1.0351
	1.7742	0.7497	1.0119

[a] Not used in fitting parameters.

scale, I. The composition of the mixtures is given as the fraction of the ionic strength contributed by $Ba(ClO_4)_2$, y_B. The observed osmotic coefficients, ϕ_{obsd} were calculated from the isopiestic concentrations and the osmotic coefficients of the reference materials as noted above. All reported values are the average of duplicate measurements.

Treatment of Results

The results were analyzed by using the neutral-electrolyte treatment. This approach is based on Scatchard's 1961 paper

Table VI. Parameters for the Two-Component Systems

	HClO^a	$Ba(ClO_4)_2$
a	1.5	1.673
$a(1)$	0.2400	0.04414
$a(2)$	0.03584	0.004816
$a(3)$	−0.001946	−0.000356
$a(4)$	0.0000440	0.0000070
$\sigma(\phi)$	0.0025	0.0033
max I, mol/kg	9.8	19.0

[a] Taken from Table 3 of ref 2.

Table VII. Osmotic and Activity Coefficients for $Ba(ClO_4)_2$

m, mol/kg	ϕ	$\log \gamma_\pm$	m, mol/kg	ϕ	$\log \gamma_\pm$
0.1	0.8575	−0.2789	1.4	1.1001	−0.2683
0.2	0.8630	−0.3190	1.6	1.1444	−0.2420
0.3	0.8770	−0.3360	1.8	1.1889	−0.2142
0.4	0.8938	−0.3431	2.0	1.2334	−0.1853
0.5	0.9121	−0.3446	2.5	1.3430	−0.1099
0.6	0.9313	−0.3425	3.0	1.4480	−0.0330
0.7	0.9511	−0.3378	3.5	1.5461	0.0428
0.8	0.9714	−0.3313	4.0	1.6358	0.1160
0.9	0.9922	−0.3232	4.5	1.7164	0.1856
1.0	1.0133	−0.3139	5.0	1.7878	0.2510
1.2	1.0563	−0.2925	5.5	1.8512	0.3125

(11) and was discussed in our previous publications (1, 2) and also in publications describing other results obtained in this laboratory (12, 13). In this approach the composition is expressed in terms of the ionic strength fraction of the component electrolytes ($HClO_4$ and $Ba(ClO_4)_2$) and the total ionic strength of the mixture. The excess free energy is expressed as a sum of contributions from the limiting two-component systems and a contribution from the three-component system. Equations for the osmotic coefficient and activity coefficients are obtained by appropriate differentiation of the excess free energy equation. The resulting equations are given in the initial paper in this series (1). The value used for the Debye–Hückel limiting slope (S) was −1.17082.

Discussion

$HClO_4$. Solutions of $HClO_4$ alone were included in several of the equilibration runs as a check on previous results. The results are shown in Table III. The parameters for $HClO_4$ derived from earlier investigations are given in Table VI. Comparison of the observed osmotic coefficients shown in Table III with values calculated by using the parameters in Table VI gives an average deviation of 0.0067. This was considered sufficiently close agreement to warrant use of the older parameters (Table VI) without modification.

$Ba(ClO_4)_2$. The observed osmotic coefficients for $Ba(ClO_4)_2$ are given in Tables I and II and the parameters derived from these data are given in Table VI. The osmotic coefficients observed in this investigation differ significantly, within the concentration range covered, from those of Robinson et al. (8, 14). The maximum deviation is 0.10 at $I = 17$ (Robinson's values are consistently higher) and the deviation is roughly proportional to the concentration. At high molalities the water fraction, which is obtained by difference, is low and therefore is quite sensitive to small uncertainties in solute molality. Thus, the effects of analytical uncertainty are magnified at high molalities. Considerable effort was devoted to an attempt to resolve this difference. The $Ba(ClO_4)_2$ stock solution was reanalyzed by several methods and the concentration carefully evaluated. Other sources for the osmotic coefficient of the reference materials were investigated. No satisfactory explanation could be found. Because of the careful analysis of the stock soltuion by several methods and over a period of several years the author has a high degree of confidence in the concentration of the stock solution and subsequent solutions prepared from it. Consequently, the author believes the results reported in this paper to be more accurate than previously published results. Osmotic and activity coefficients calculated from the parameters in Table VI at integral values of molality are given in Table VII.

Table VIII. Parameters for the Three-Component System $HClO_4$–$Ba(ClO_4)_2$–H_2O

$b(01)$	−0.0403	$b(12)$	−0.0028
$b(02)$	−0.0234	$b(13)$	−0.00031
$b(03)$	0.00029	$\sigma(\phi)$	0.0046

Table IX. Values of the Osmotic and Activity Coefficients for the System $HClO_4$ (A)–$Ba(ClO_4)_2$ (B)–H_2O

			$\log \gamma_\pm$	
I, mol/kg	y_B	ϕ	A	B
1.0	0.0	1.0442	−0.0880	−0.2729
	0.2	1.0204	−0.0997	−0.2866
	0.4	0.9941	−0.1112	−0.3002
	0.6	0.9641	−0.1223	−0.3134
	0.8	0.9281	−0.1332	−0.3264
	1.0	0.8823	−0.1437	−0.3392
2.0	0.0	1.2133	0.0205	−0.1880
	0.2	1.1674	−0.0080	−0.2202
	0.4	1.1199	−0.0352	−0.2514
	0.6	1.0690	−0.0614	−0.2817
	0.8	1.0122	−0.0864	−0.3111
	1.0	0.9444	−0.1104	−0.3396
4.0	0.0	1.6253	0.3165	0.0681
	0.2	1.5159	0.2430	−0.0072
	0.4	1.4108	0.1743	−0.0791
	0.6	1.3075	0.1102	−0.1478
	0.8	1.2016	0.0506	−0.2136
	1.0	1.0854	−0.0046	−0.2767
6.0	0.0	2.1039	0.6730	0.3617
	0.2	1.9074	0.5412	0.2401
	0.4	1.7279	0.4206	0.1256
	0.6	1.5614	0.3106	0.0171
	0.8	1.4007	0.2107	−0.0862
	1.0	1.2334	0.1204	−0.1852
8.0	0.0	2.6234	1.0671	0.6557
	0.2	2.3185	0.8658	0.4900
	0.4	2.0513	0.6849	0.3353
	0.6	1.8149	0.5232	0.1896
	0.8	1.5977	0.3797	0.0504
	1.0	1.3786	0.2532	−0.0843

$HClO_4$–$Ba(ClO_4)_2$ Mixtures. Values of the b parameters for this three-component system are given in Table VIII. Data points falling beyond the maximum concentration used to derive the two-component data, as given in Table VI, were not used in deriving the parameters in Table VIII. These points are identified in Table V. Fits to the data were obtained by using fewer than five b parameters; in all cases the standard deviation was significantly higher.

As with the previous systems investigated, it is apparent that the equations given by Scatchard provide an adequate and convenient means of representing the osmotic coefficients of three-component mixtures. This is an especially severe test for these equations since, as noted above, the osmotic coefficients are quite different at high concentrations. The Scatchard equations also provide a simple method for calculating the activity coefficients that is not dependent on an integration to zero concentration, as is the case with the McKay–Perring method (15). Values for the osmotic coefficient and the activity coefficients for various mixture compositions are given in Table IX.

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Glossary

A	symbols subscripted A refer to $HClO_4$
B	symbols subscripted B refer to $Ba(ClO_4)_2$
m	molal concentration, mol/kg
I	total ionic strength, mol/kg
y	ionic strength fraction
ϕ	molal osmotic coefficient
$\gamma_\pm$	mean molal activity coefficient
a, $a(1)$, $a(2)$, $a(3)$, $a(4)$	parameters in Scatchard equations for single electrolytes, see ref *1*
$b(01)$, $b(02)$, $b(03)$, $b(12)$, $b(13)$	parameters in Scatchard equations for electrolyte mixtures, see ref *1*
$\sigma(\phi)$	standard deviation for osmotic coefficients

Registry No. $HClO_4$, 7601-90-3; $Ba(ClO_4)_2$, 13465-95-7.

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Excess Volumes of *n*-Octane + *n*-Undecane between 288.15 and 308.15 K

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Excess molar volumes of *n*-octane + *n*-undecane have been determined from density measurements at 288.15, 293.15, 298.15, 303.15, and 308.15 K, and an approximation to $(\partial V_m^E/\partial T)_p$ in the neighborhood of 298.15 K has been calculated.

Introduction

This article reports excess volumes of $\{xn\text{-}C_8H_{18} + (1 - x)C_{11}H_{24}\}$ determined from density measurements at 288.15, 293.15, 298.15, 303.15, and 308.15 K for $0 \leq x \leq 1$. Three-parameter empirical equations have been fitted to the results for each temperature. By fitting a quadratic in T to the five values obtained for each parameter, an approximate expression for $(\partial V_m^E/\partial T)_p$ in the neighborhood of 298.15 K has been obtained.

Experimental Section

Densities were measured with an Anton Paar D.M.A. 60/602 densimeter with resolution of $\pm 1.5 \times 10^{-6}$ g·cm^{-3}. The temperature of the water bath containing the oscillator tube was kept constant to within ±0.005 K with a Hetofrig theromostat. The operational technique has been described elsewhere (*1*). The *n*-octane (Fluka purum >99 mol %) and *n*-undecane (Merck, >99 mol %) were carefully dried with molecular sieves (Union Carbide type 4A, from Fluka) and used directly without further purification. The densities of *n*-octane and *n*-undecane at the various temperatures employed are given in Table I along with values at 293.15 and 298.15 K from the literature for comparison (*2*, *3*).

Table I. Experimental Densities (g•cm^{-3}) at Various Temperatures and Comparison with Literature Values

	T/K				
	288.15	393.15	298.15	303.15	308.15
	Experimental				
n-octane	0.70660	0.70256	0.69853	0.69448	0.69042
n-undecane	0.74429	0.74059	0.73690	0.73320	0.72951
	Literature				
n-octane		0.70252[a]	0.69849[a]		
n-undecane		0.74017[b]			

[a] Reference *2*. [b] Reference *3*.

Results and Discussion

Table II lists the excess molar volumes V_m^E calculated from the densities for each x and T. For each T, equations of the form

$$V_m^E/(\text{cm}^3\cdot\text{mol}^{-1}) = x(1 - x) \sum_{k \leq 2} A_k(2x - 1)^k \qquad (1)$$

were fitted (*4*) to the experimental results. The parameters A_k and standard deviations of fit are listed in Table III. Figure 1 shows the fitted V_m^E curves.

Once the A_k has been estimated, quadratics in T were fitted

$$A_k(T) = A_{k0} + A_{k1}(T/k) + A_{k2}(T/k)^2 \qquad (2)$$

The values so obtained for the A_{kj} are listed in Table IV. Equations 1 and 2 allow an approximation to $(\partial V_m^E/\partial T)_p$ to be

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