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Vapor−Liquid Equilibria in the Systems 2,2′-Oxybis[propane] + 1-Chlorobutane and 2,2′-Oxybis[propane] + 1-Chlorobutane + Cyclohexane

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Vapor−liquid equilibrium at 101.3 kPa has been determined for the binary system 2,2′-oxybis[propane] + 1-chlorobutane and the ternary system 2,2′-oxybis[propane] + 1-chlorobutane + cyclohexane. The binary 2,2′-oxybis[propane] + 1-chlorobutane exhibits slight deviations from ideality. The data were correlated by the Redlich−Kister and Wisniak−Tamir equations, and the appropriate parameters are reported. The activity coefficients of the ternary system can be predicted from those of the pertinent binary systems. No ternary azeotrope is present.

The present work was undertaken to measure vapor−liquid equilibrium (VLE) data for the title systems for which no isobaric data are available. Data for the binary systems 2,2′-oxybis[propane] + cyclohexane and 1-chlorobutane + cyclohexane have already been reported at 101.3 kPa (Wisniak, 1995a,b). Both systems exhibit moderate positive deviations from ideal behavior. The binary system 1-chlorobutane + cyclohexane presents an azeotrope that boils at 350.01 K and contains 57.5 mol% 1-chlorobutane. The related system 1,1′-oxybis[propane] + 1-chlorobutane has been studied at 298.15 K (Lepori et al., 1991) and shown to behave almost ideally, with infinite dilution activity coefficients of 1.05 for both components.

Experimental Section

Purity of Materials. 2,2′-Oxybis[propane] (99.9+ mol %) was purchased from Aldrich, and 1-chlorobutane (99.84+ mol %) and cyclohexane (99.5+ mol %) were purchased from Merck. The reagents were used without further purification after gas chromatography failed to show any significant impurities. The properties and purity (as determined by GLC) of the pure components appear in Table 1.

Apparatus and Procedure. An all-glass modified Dvorak and Boublik recirculation still (Boublikova and Lu, 1969) was used in the VLE measurements. The experimental features have been described in a previous paper (Wisniak and Tamir, 1975). All analyses were carried out by gas chromatography on a Gow-Mac Series 550P apparatus provided with a thermal conductivity detector and a Spectra Physics Model SP 4290 electronic integrator. The column was 3 m long and 0.2 cm in diameter and was packed with SE-30 and operated at 319.15 K; injector and detector temperatures were 493.15 and 543.15 K, respectively. Very good separation for the binary and ternary systems was achieved under these conditions, and calibration analyses were carried out to convert the peak ratio to the mass composition of the sample. Concentration measurements were accurate to better than ±0.008 mole fraction unit. The accuracy in the determination of pressure P and temperature T was at least ±0.1 kPa and 0.02 K, respectively.

Table 1. Mole Percent GLC Purities, Refractive Index n_D at Na D Line, and Normal Boiling Points T of the Pure Components

component (purity, mol %)	n_D(298.15 K)	T/K
2,2′- oxybis[propane] (99.9)	1.3654[a]	341.55[a]
	1.3655[b]	341.45[b]
	1.3665[b]	341.45[b]
1-chlorobutane (99.84)	1.3999[a]	351.58[a]
	1.4000[b]	351.58[b]
cyclohexane (99.5)	1.4233[a]	353.84[a]
	1.42354[b]	353.888[b]

[a] Measured. [b] TRC (1974).

Table 2. Experimental Vapor−Liquid Equilibrium Data for 2,2′-Oxybis[propane] (1) + 1-Chlorobutane (2) at 101.3 kPa

T/K	x_1	y_1	γ_1	γ_2	$-B_{11}$/ (cm^{-3}/ mol^{-1})	$-B_{22}$/ (cm^{-3}/ mol^{-1})	$-B_{12}$/ (cm^{-3}/ mol^{-1})
351.58	0	0					
350.98	0.035	0.050	1.0605	1.0034	952	647	736
350.82	0.049	0.069	1.0574	1.0023	953	648	737
350.25	0.078	0.107	1.0552	1.0087	956	650	740
349.99	0.100	0.136	1.0517	1.008	958	652	741
349.40	0.143	0.190	1.0478	1.0101	962	654	744
349.23	0.165	0.217	1.0399	1.0077	963	655	745
348.90	0.190	0.246	1.0360	1.0099	965	657	746
348.09	0.263	0.315	0.9810	1.0338	970	660	750
347.23	0.335	0.395	0.9888	1.0402	975	664	754
345.64	0.502	0.569	0.9958	1.0411	985	672	762
345.05	0.564	0.629	0.9977	1.0427	989	675	765
344.56	0.612	0.674	1.0003	1.0448	992	677	767
344.05	0.668	0.724	0.9989	1.0530	995	680	770
343.75	0.715	0.765	0.9955	1.0538	997	681	771
343.50	0.743	0.789	0.9960	1.0563	999	682	773
343.07	0.816	0.851	0.9910	1.0569	1002	685	775
342.74	0.855	0.884	0.9924	1.0558	1004	686	777
342.57	0.871	0.897	0.9941	1.0567	1005	687	778
342.21	0.904	0.925	0.9977	1.0559	1008	689	780
341.97	0.930	0.946	0.9989	1.0576	1009	690	781
341.45	1	1					
γ^∞ [a]			1.053	0.9952			

[a] Calculated according to the method of Wisniak et al. (1995).

Results

The temperature T and liquid-phase x_i and vapor-phase y_i mole fraction measurements at P = 101.3 kPa are reported in Tables 2 and 3 and Figure 1, together with the activity coefficients γ_i which were calculated from the

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Table 3. Experimental Vapor–Liquid Equilibrium Data for 2,2′-Oxybis[propane] (1) + 1-Chlorobutane (2) + Cyclohexane (3) at 94.4 kPa

					activity coefficients								
T/K	x_1	x_2	y_1	y_2	γ_1	γ_2	γ_3	$-B_{12}$/ (cm^3·mol^{-1})	$-B_{13}$/ (cm^3·mol^{-1})	$-B_{23}$/ (cm^3·mol^{-1})	$-B_{11}$/ (cm^3·mol^{-1})	$-B_{22}$/ (cm^3·mol^{-1})	$-B_{33}$/ (cm^3·mol^{-1})
342.45	0.891	0.054	0.911	0.044	0.9907	1.0853	1.1511	1353	1244	1204	1306	1286	1223
342.95	0.805	0.034	0.840	0.029	0.9970	1.0983	1.1323	1349	1239	1200	1301	1282	1219
343.55	0.730	0.135	0.773	0.115	0.9923	1.0848	1.1269	1342	1234	1194	1295	1275	1213
343.55	0.710	0.202	0.758	0.167	0.9983	1.0541	1.1684	1342	1233	1194	1295	1275	1213
343.75	0.703	0.054	0.757	0.046	1.0016	1.0904	1.0928	1340	1232	1192	1293	1273	1211
343.95	0.717	0.052	0.765	0.045	0.9869	1.1040	1.1047	1338	1230	1190	1291	1272	1209
344.05	0.646	0.308	0.710	0.252	1.0141	1.0280	1.1108	1337	1229	1190	1290	1271	1209
344.05	0.666	0.168	0.719	0.141	0.9955	1.0576	1.1280	1337	1229	1190	1290	1271	1209
344.15	0.537	0.322	0.601	0.275	1.0280	1.0669	1.1905	1336	1228	1189	1289	1270	1208
345.55	0.486	0.448	0.558	0.383	1.0131	1.0273	1.1515	1323	1217	1177	1277	1257	1196
345.55	0.483	0.154	0.538	0.143	0.9833	1.1111	1.1228	1322	1216	1177	1276	1257	1196
346.15	0.396	0.421	0.460	0.370	1.0064	1.0357	1.1640	1317	1211	1172	1271	1252	1191
346.35	0.402	0.524	0.471	0.456	1.0112	1.0200	1.2168	1315	1210	1170	1269	1250	1189
346.85	0.313	0.516	0.365	0.471	0.9904	1.0539	1.1842	1310	1206	1166	1265	1246	1185
346.95	0.360	0.183	0.420	0.177	0.9877	1.1142	1.0841	1309	1205	1166	1264	1245	1184
347.15	0.282	0.471	0.325	0.441	0.9666	1.0702	1.1569	1307	1203	1163	1262	1243	1182
347.25	0.332	0.613	0.403	0.544	1.0190	1.0124	1.1753	1307	1202	1163	1261	1242	1182
347.25	0.319	0.359	0.368	0.335	0.9701	1.0655	1.1202	1307	1202	1163	1261	1242	1182
347.35	0.261	0.466	0.315	0.428	1.0082	1.0461	1.1405	1305	1201	1162	1260	1241	1181
347.45	0.263	0.374	0.308	0.354	0.9762	1.0692	1.1270	1304	1200	1161	1259	1240	1180
347.65	0.271	0.271	0.330	0.263	1.0130	1.0922	1.0676	1303	1199	1160	1258	1239	1178
347.65	0.241	0.305	0.296	0.296	1.0144	1.0904	1.0802	1302	1198	1159	1257	1238	1178
347.95	0.222	0.584	0.259	0.549	0.9583	1.0496	1.1794	1300	1196	1157	1255	1236	1176
347.95	0.247	0.686	0.309	0.623	1.0282	1.0122	1.2004	1299	1196	1157	1254	1235	1176
348.05	0.186	0.537	0.222	0.505	0.9761	1.0469	1.1677	1298	1195	1156	1254	1235	1175
348.05	0.196	0.368	0.247	0.353	1.0327	1.0646	1.0880	1298	1195	1156	1253	1234	1175
348.15	0.144	0.53	0.178	0.503	1.0143	1.0523	1.1545	1298	1194	1155	1253	1234	1174
348.25	0.163	0.477	0.203	0.455	1.011	1.0512	1.1227	1296	1193	1154	1252	1233	1173
348.45	0.179	0.661	0.226	0.615	1.0189	1.0206	1.1652	1294	1191	1153	1250	1231	1171
348.65	0.124	0.368	0.148	0.371	0.9606	1.0976	1.1043	1293	1190	1151	1248	1229	1170
348.75	0.157	0.258	0.194	0.265	0.9933	1.1219	1.0754	1292	1190	1151	1248	1229	1170
348.75	0.148	0.451	0.177	0.438	0.9613	1.0585	1.1129	1292	1189	1150	1247	1228	1169
348.75	0.082	0.575	0.103	0.555	1.0088	1.0507	1.1563	1292	1189	1150	1247	1228	1169
348.85	0.162	0.713	0.204	0.666	1.0120	1.0132	1.2006	1291	1189	1150	1247	1228	1168
348.85	0.114	0.620	0.144	0.587	1.0103	1.0270	1.1721	1291	1188	1150	1247	1227	1168
349.15	0.098	0.674	0.127	0.639	1.0278	1.0204	1.1818	1289	1186	1148	1244	1225	1166
349.35	0.097	0.300	0.122	0.315	0.9951	1.1226	1.0649	1286	1184	1146	1242	1223	1164
349.35	0.128	0.772	0.166	0.724	1.0181	1.0017	1.2556	1286	1184	1145	1242	1223	1164
349.45	0.151	0.145	0.195	0.161	1.0162	1.1809	1.0415	1285	1184	1145	1241	1222	1163
349.45	0.059	0.425	0.072	0.430	0.9705	1.0781	1.0966	1285	1184	1145	1241	1222	1163
349.55	0.035	0.600	0.046	0.585	1.0227	1.0362	1.1465	1284	1183	1144	1240	1221	1162
349.65	0.075	0.759	0.098	0.720	1.0272	1.0053	1.2397	1284	1182	1143	1240	1221	1162
349.75	0.047	0.340	0.059	0.362	0.9756	1.1264	1.0672	1283	1181	1143	1239	1220	1161
349.85	0.094	0.831	0.128	0.789	1.0623	1.0007	1.2419	1282	1181	1142	1238	1219	1160
349.85	0.083	0.838	0.117	0.792	1.0997	0.9964	1.2852	1282	1180	1142	1238	1219	1160
349.95	0.101	0.205	0.127	0.224	0.9761	1.1491	1.0482	1281	1179	1141	1237	1218	1159
349.95	0.123	0.122	0.159	0.137	1.0033	1.1832	1.0432	1280	1179	1141	1237	1218	1159
350.05	0.058	0.807	0.078	0.772	1.0366	1.0029	1.2415	1280	1179	1140	1236	1217	1159
350.05	0.073	0.241	0.096	0.264	1.0155	1.1438	1.0421	1279	1178	1140	1236	1217	1158
350.35	0.139	0.060	0.186	0.069	1.0251	1.2040	1.0310	1277	1176	1138	1234	1215	1156
350.55	0.057	0.191	0.075	0.217	0.9986	1.1713	1.0371	1275	1174	1136	1232	1213	1154
351.05	0.044	0.158	0.060	0.184	1.0075	1.1845	1.0296	1271	1171	1132	1227	1209	1151
351.55	0.035	0.128	0.049	0.155	1.0414	1.2156	1.0177	1266	1167	1129	1223	1205	1147
351.85	0.029	0.090	0.043	0.115	1.1010	1.2636	1.0144	1263	1164	1126	1221	1202	1144
352.25	0.022	0.068	0.033	0.088	1.0908	1.2645	1.0142	1260	1161	1123	1217	1199	1141
352.55	0.025	0.076	0.036	0.097	1.0284	1.2371	1.0021	1257	1158	1120	1214	1196	1139

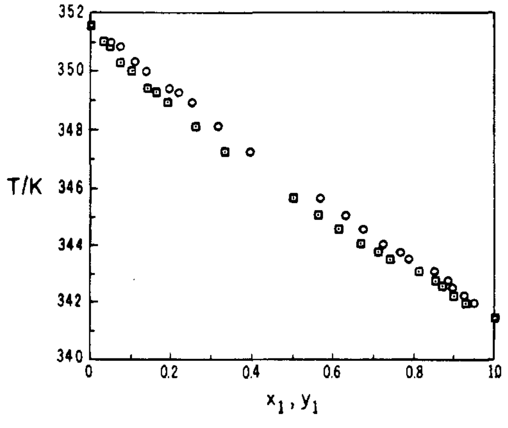

Figure 1. Boiling point diagram at 101.3 kPa for the system 2,2′-oxybis[propane] (1) + 1-chlorobutane (2).

following equation (Van Ness and Abbott, 1982):

$$\ln \gamma_i = \ln(Py_i/P_i^\circ x_i) + (B_{ii} - v_i^{\mathrm{L}})(P - P_i^\circ)/RT + (P/2RT)\sum_1^n\sum_1^n y_j y_k(2\delta_{ji} - \delta_{jk}) \quad (1)$$

where

$$\delta_{ji} = 2B_{ji} - B_{jj} - B_{ii} \quad (2)$$

The standard state for the calculation of activity coefficients is the pure component at the pressure and temperature of the solution. The pure component vapor pressures P_i° were calculated according to the Antoine equation

$$\log(P_i^\circ)/\mathrm{kPa} = A_i - B_i/(T/\mathrm{K} - C_i) \quad (3)$$

The constants A_i, B_i, and C_i are reported in Table 4. The

Table 4. Antoine Coefficients, Eq 3

compound	A_i	B_i	C_i
2,2′- oxybis[propane][a]	6.222 00	1257.60	43.14
1-chlorobutane[b]	6.062 75	1227.429	49.05
cyclohexane[b]	5.964 07	1200.31	50.65

[a] Yaws (1992). [b] TRC (1974).

molar virial coefficients B_{ii} and B_{ij} were estimated according to the method of O'Connell and Prausnitz (1967) using the molecular parameters suggested by the authors and assuming the association parameter η to be zero. The last two terms in eq 1 contributed less than 2% to the activity coefficient, and their influence was important only at very dilute concentrations. The calculated activity coefficients are reported in Tables 2 and 3 and are estimated accurate to within ±3%. Table 2 also contains the activity coefficients at infinite dilution calculated by the method suggested by Wisniak et al. (1995). As seen from Table 2 the binary system 2,2′- oxybis[propane] + 1-chlorobutane exhibits slight deviations from ideality, its behavior being very similar to that of the binary system 1,1′-oxybis[propane] + 1 chlorobutane (Lepori et al., 1991).

The binary data reported in Table 2 were found to be thermodynamically consistent by the area test (Van Ness and Abbott, 1982) and the L−W method of Wisniak (1993). The ternary activity coefficients reported in Table 3 were found to be thermodynamically consistent as tested by the L−W method of Wisniak (1993) and the McDermot−llis method (1965) modified by Wisniak and Tamir (1977). According to these references two experimental points, a and b are considered thermodynamically consistent if the following condition is fulfilled:

$$D < D_{\max} \qquad (4)$$

The local deviation D is given by

$$D = \sum_{i=1}^{N} (x_{ia} + x_{ib})(\ln \gamma_{ia} - \ln \gamma_{ib}) \qquad (5)$$

where N is the number of components and the maximum deviation $D_{\max}$ is

$$D_{\max} = \sum_{i=1}^{N} (x_{ia} + x_{ib})\left(\frac{1}{x_{ia}} + \frac{1}{y_{ia}} + \frac{1}{x_{ib}} + \frac{1}{y_{ib}}\right)\Delta x + 2\sum_{i=1}^{N} |\ln \gamma_{ib} - \ln \gamma_{ia}|\Delta x + \sum_{i=1}^{N} (x_{ia} + x_{ib})\frac{\Delta P}{P} + \sum_{i=1}^{N} (x_{ia} + x_{ib})\{(t_a + C_i)^{-2} + (t_b + C_i)^{-2}\}\Delta T \qquad (6)$$

The errors in the measurements Δx, ΔP, and Δt were as previously indicated. The first term in eq 6 was the dominant one. For the experimental points reported here D never exceeded 0.060 while the smallest value of $D_{\max}$ was 0.374.

The activity coefficients for the ternary system were correlated by the following Redlich−Kister expansion (Hala et al., 1967):

$$\ln \gamma_1/\gamma_2 = b_{12}(x_2 - x_1) - c_{12}[(x_1 - x_2)^2 - 2x_1x_2] + d_{12}(x_2 - x_1)[(x_1 - x_2)^2 - 4x_1x_2] + x_3[b_{13} + c_{13}(2x_1 - x_3) + d_{13}(x_1 - x_3)(3x_1 - x_3) - b_{23} - c_{23}(2x_2 - x_3) - d_{23}(x_2 - x_3)(3x_2 - x_3) + C_1(x_2 - x_1)] \qquad (7)$$

Table 5. Constants for the Redlich−Lister Model, Eq 7

A. Binary Systems

system	b_{ij}	c_{ij}	d_{ij}	rmsd	% dev[a]	max % dev[b]
2,2′-oxybis[propane] (1) + 1-chlorobutane (2)	0.0250	−0.0102	0	0.007	2.7	7.9
2,2′-oxybis[propane] (1) + cyclohexane (3)[c]	0.0654	−0.0041	0	0.004	1.8	3.2
1-chlorobutane (2) + cyclohexanes (3)[d]	0.1320	−0.0085	0.0160	0.003	2.4	3.7

B. Ternary System

											γ_1/γ_2			γ_1/γ_3	
	b_{12}	c_{12}	d_{12}	b_{13}	c_{13}	d_{13}	b_{23}	c_{23}	d_{23}	C_1	rmsd	max % dev	% dev	rmsd	max % dev	% dev
2,2′-oxybis[propane] (1)	0.0250	−0.0102	0	0.0654	−0.0041	0	0.1320	−0.0085	0.0160	0	0.004	7.9	2.6	0.006	9.7	4.1
+ 1-chlorobutane (2)																
+ cyclohexane (3)										−0.06074	0.004	7.6	2.6	0.005	7.6	3.2

[a] Percent average deviation. [b] Maximum percent deviation. [c] Wisniak (1995a). [d] Wisniak (1995b).

Table 6. Coefficients in the Correlation of Boiling Points, Eq 8, and Root Mean Square Deviations in Temperature, rmsd(*T*/K)

Binary Systems

system	C_0	C_1	C_2	rmsd	% dev[a]
2,2′-oxybis[propane] (1) + 1-chlorobutane (2)	−3.909 20	2.549 75		0.02	0.10
2,2′-oxybis[propane] (1) + cyclohexane (3)[b]	−6.789 85	3.003 82		0.02	0.08
1-chlorobutane (2) + cyclohexane (3)[c]	−10.537 7	1.932 12	−2.154 86	0.01	0.02

Ternary System

	A	*B*	*C*	rmsd	% dev[a]
2,2′-oxybis[propane] (1) + 1-chlorobutane (2) + cyclohexane (3)	−7.048 17			0.03	0.3

[a] Average percent deviation. [b] Wisniak (1995a). [c] Wisniak (1995b).

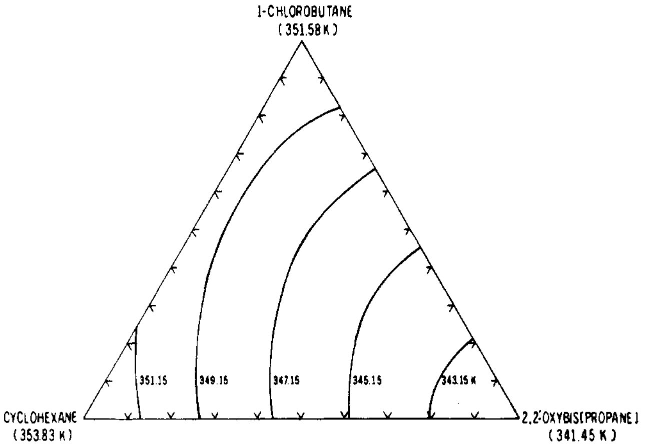

Figure 2. Isothermals for the ternary system 2,2′-oxybis[propane] (1) + 1-chlorobutane (2) + cyclohexane (3) at 101.3 kPa, calculated with eq 8 and the constants in Table 6.

where b_{ij}, c_{ij}, and d_{ij} are constants for the pertinent binary and C_1 is a ternary constant. The equations for two other pairs of activity coefficients were obtained by cyclic rotation of the indices. All the constants in eq 7 are assumed to be independent of the temperature. Data for the binary systems 2,2′-oxybis[propane] + cyclohexane and 1-chlorobutane + cyclohexane have already been reported (Wisniak, 1995a,b). The ternary Redlich–Kister coefficient was obtained by a Simplex optimization technique. The differences between the values of the root mean square deviation for the activity coefficient for the two cases—with and without the ternary constant C_1 (Table 5)—are statistically not significant, suggesting that ternary data can be predicted directly from the binary systems.

The boiling points of the systems were correlated by the equation proposed by Wisniak and Tamir (1976):

$$T/\mathrm{K} = \sum_{i=1}^{n} x_i T_i^{\circ}/\mathrm{K} + \sum_{i,j=1}^{n} \{x_i x_j \sum_{k=0}^{m} C_k (x_i - x_j)^k\} + x_1 x_2 x_3 \{A + B(x_1 - x_2) + C(x_1 - x_3) + D(x_2 - x_3)\} \quad (8)$$

In this equation n is the number of components ($n = 2$ or 3), T_i° is the boiling point of the pure component i, and $m + 1$ is the number of terms in the series expansion of $x_i - x_j$. C_k are the binary constants, and A, B, C, and D are ternary constants. An equation of the same structure can be used for the direct correlation of ternary data, without use of binary data. Both forms will require about the same number of constants for similar accuracy, but the direct correlation allows an easier calculation of boiling isotherms (Figure 2). The various constants of eq 8 are reported in Table 6, which also contains information indicating the degree of goodness of the correlation.

Glossary

A_i, B_i, C_i	Antoine constants, eq 3
B_{ii}, B_{ij}	second molar virial coefficients, eqs 1 and 2
b_{ii}, c_{ij}, d_{ii}	Redlich–Kister constants, eq 7
C_k	constants, eq 8
N	number of measurements
P	total pressure
P_i°	vapor pressure of pure component i
R	gas constant
rmsd(T)	root mean square deviation, $\{\sum(T_{\mathrm{exptl}} - T_{\mathrm{calcd}})^2\}^{0.5}/N$
rmsd(γ_i)	root mean square deviation, $\{\sum(\gamma_{i,\mathrm{expt}} - \gamma_{i,\mathrm{calcd}})^2\}^{0.5}/N$
T	boiling temperature of a mixture
T_i°	boiling temperature of pure component i
v_i^L	molar volume of liquid component i
x_i, y_i	mole fraction of component i in the liquid and vapor phases
γ_i	activity coefficient of component i
δ_{ij}	molar virial coefficient parameter, eq 2

Subscripts

exptl	experimental value
calcd	calculated value
i	component i

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