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桉叶油素与醇类在298.15，308.15 K的过量体积*

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摘 要：在298.15 K和308.15 K下用振动管密度计在全浓度范围内测量了桉叶油素分别与甲醇、乙醇、正丙醇、异丙醇、正丁醇、异丁醇构成的二元混合物的过量体积．这6个体系的过量体积 V^E 在全浓度范围内均为负值．V^E 值用Redlich-Kister方程进行拟合．

关键词：桉叶油素；过量体积；振动管密度计
中图分类号：O642.42 **文献标识码**：A **文章编号**：0529-6579（2001）04-0076-05

液体与液体混合物的热力学性质和传递性质，可用于了解液体和液体混合物组分分子间的相互作用，也用于传热、传质、流体流动等方面的研究．桉叶油素（1，8-cineole，$C_{10}H_{18}O$）在医药、日用化工等方面有着广泛的用途．在研究从桉叶油中分离提取桉叶油素时，本文报道在298.15 K和308.15 K下桉叶油素与甲醇、乙醇、正丙醇、异丙醇、正丁醇、异丁醇构成6个二元系的 V^E 数据．

1 实验部分

1.1 试剂

甲醇、乙醇、正丙醇、异丙醇、正丁醇、异丁醇均用国产分析纯试剂经高效精馏柱精馏，取中间馏分再用分子筛干燥；桉叶油素用特殊精馏法从桉叶油中提取，再用高效精馏柱精馏，经PE Autosystem XL气相色谱分析，桉叶油素纯度（x）为99.67%，甲醇、乙醇、正丙醇、异丙醇、正丁醇、异丁醇纯度（x）均大于99.93%，试剂存放于干燥器中备用．各试剂在298.15 K和308.15 K时的密度，用Anton Paar DMA4500型振动管密度计测定，与文献值同列于表1．

1.2 实验方法

桉叶油素与甲醇、乙醇、正丙醇、异丙醇、正丁醇、异丁醇6个二元系在298.15，308.15 K的过量体积用Anton Paar DMA 4500型振动管密度计测定[9]，仪器内置恒温精度为±0.01 K．测定前用干燥空气和二次蒸馏水校正，精确至 1×10^{-5} g/cm^3，误差为 $\pm5\times10^{-5}$ g/cm^3．空气与水的密度测定值（文献值），在298.15 K时分别为0.001 19（0.001 183[10]），0.997 05（0.997 044[10]）；在308.15 K时分别为0.001 15（0.001 145 8[10]），0.994 03（0.994 06[8]）g/cm^3．为防止挥发用10 mL带塞磨口试管配制溶液，溶液配制用称量法，天平称准至0.000 1 g，浓度误差为±0.000 1．

表1 纯液体在298.15和308.15 K的密度和折射率

Tab.1 Densities and refractive index of pure liquids at 298.15 and 308.15 K

纯液体	ρ/(g·cm^{-3},298.15 K)		ρ/(g·cm^{-3},308.15 K)	n_D^{20}	
	本文值	文献值	本文值	本文值	文献值
甲醇	0.78658	0.78656[1]	0.77709	1.3287	1.3288[7]
乙醇	0.78509	0.78512[2]	0.77661	1.3611	1.3611[7]
正丙醇	0.79952	0.79954[3]	0.79140	1.3850	1.3850[7]
异丙醇	0.78089	0.78085[4]	0.77227	1.3771	1.3772[8]
正丁醇	0.80571	0.80575[5]	0.81028	1.3993	1.3993[8]
异丁醇	0.79782	0.79780[5]	0.78998	1.3958	1.3958[8]
桉叶油素	0.91995	0.92029[6]	0.91137	1.4573	1.4575[7]

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1.3 实验结果

将测定的各体系二元液体混合物和纯液体的密度按下式[11]计算过量体积 V^E

$$V^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \frac{x_1 M_1}{\rho_1} - \frac{x_2 M_2}{\rho_2} \qquad (1)$$

并用下列方程[11]拟合：

$$V^E = x(1-x)\sum_{i=0}^{k} A_i(1-2x)^i \qquad (2)$$

V^E 实验值 V^E_{exp} 的标准偏差 σ 由以下方程计算：

$$\sigma = \left[\sum_{l}^{N}(V^E_{exp} - V^E_{cal}\Big/(N-n)\right]^{1/2} \qquad (3)$$

式中，x_1, x_2 为摩尔分数，M_1, M_2 为摩尔质量，ρ 为溶液的密度，ρ_1、ρ_2 分别为纯物质的密度，A_i 为方程(2)拟合参数；V^E_{exp} 为过量体积实验值，V^E_{cal} 为方程(2)的计算值，N 为实验数据组数，n 为回归方程(2)参数的数目.

各体系二元混合物的密度、过量体积的实验值列于表 2；图 1 为 298.15 K 和 308.15 K 时 6 个二元混合物过量体积的实验值及拟合的曲线；各体系的拟合参数和标准偏差列于表 3.

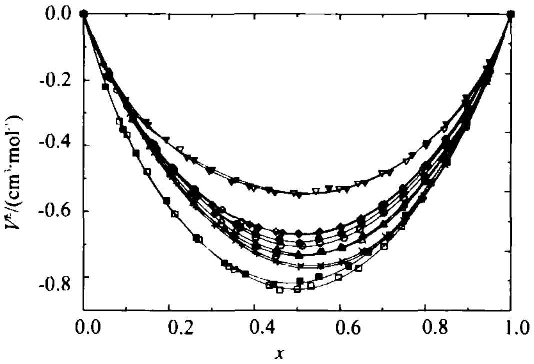

图 1　298.15,308.15 K 桉叶油素与醇类二元混合物的 $V^E - x$ 图

Fig.1　Excess molar volumes V^E for binary mixtures of 1.8-cineole + alkanols at 298.15 K and 308.15 K

298.15 K: (1 − x)(1,8-cineole) + ■ $x(CH_3OH)$; ● $x(C_2H_5OH)$; ▲ $x(CH_3(CH_2)_2OH)$; ▼ $x((CH_3)_2CHOH)$; ◆ $x(CH_3(CH_2)_3OH)$; + $x((CH_3)_2CHCH_2OH)$)

308.15 K: (1 − x)(1,8-cineole) + □ $x(CH_3OH)$; ○ $x(C_2H_5OH)$; △ $x(CH_3(CH_2)_2OH)$; ▽ $x((CH_3)_2CHOH)$; ◇ $x(CH_3(CH_2)_2OH)$; × $x((CH_3)_2CHCH_2OH)$

表 2　298.15 K 和 308.15 K 时各体系的密度、过量体积的实验值

Tab.2　Densities, experimental excess molar volumes at 298.15 and 308.15 K for various systems

x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^E_{exp} 1)	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^E_{exp}	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^E_{exp}	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^E_{exp}
(1 − x)(1,8-cineole) + $x(CH_3OH)$, 298.15 K											
0.0496	0.91954	−0.2208	0.5509	0.89664	−0.7970	0.0922	0.91880	−0.3502	0.6206	0.88968	−0.7653
0.1230	0.91811	−0.4229	0.6667	0.88401	−0.7216	0.1947	0.91618	−0.5662	0.7160	0.87693	−0.6691
0.2629	0.91393	−0.6784	0.7551	0.87022	−0.6099	0.3286	0.91124	−0.7551	0.8133	0.85822	−0.5143
0.3787	0.90876	−0.7912	0.8498	0.84895	−0.4377	0.4449	0.90489	−0.8204	0.8962	0.83460	−0.3207
0.5062	0.90042	−0.8109	0.9471	0.81458	−0.1793						
(1 − x)(1,8-cineole) + $x(CH_3OH)$, 308.15 K											
0.0833	0.91033	−0.3254	0.5320	0.88951	−0.8256	0.1019	0.90990	−0.3677	0.5959	0.88348	−0.7999
0.1489	0.90878	−0.4788	0.6445	0.87803	−0.7709	0.2144	0.90688	−0.6068	0.7030	0.87001	−0.7059
0.2692	0.90490	−0.6837	0.7441	0.86321	−0.6444	0.3404	0.90187	−0.7660	0.7926	0.85373	−0.5632
0.3536	0.90122	−0.7764	0.8507	0.83957	−0.4498	0.4344	0.89671	−0.8246	0.8922	0.82677	−0.3437
0.4586	0.89519	−0.8386	0.9444	0.80648	−0.1956	0.5005	0.89212	−0.8364			
(1 − x)(1,8-cineole) + $x(C_2H_5OH)$, 298.15 K											
0.0600	0.91804	−0.1824	0.5502	0.88513	−0.6827	0.1027	0.91640	−0.2807	0.6022	0.87898	−0.6656
0.1709	0.91339	−0.4102	0.6505	0.87247	−0.6346	0.2118	0.91134	−0.4736	0.7006	0.86487	−0.5953
0.2652	0.90844	−0.5524	0.7498	0.85632	−0.5431	0.3094	0.90573	−0.6008	0.8049	0.84514	−0.4606
0.3573	0.90246	−0.6386	0.8491	0.83482	−0.3889	0.4016	0.89914	−0.6674	0.8995	0.82107	−0.2837
0.4577	0.89439	−0.6844	0.9500	0.80473	−0.1611	0.5048	0.88998	−0.6951			
(1 − x)(1,8-cineole) + $x(C_2H_5OH)$, 308.15 K											
0.0777	0.90881	−0.2318	0.5528	0.87619	−0.6955	0.1131	0.90734	−0.3014	0.6087	0.86953	−0.6801
0.1651	0.90500	−0.3976	0.6464	0.86442	−0.6535	0.2091	0.90282	−0.4698	0.7032	0.85574	−0.6025
0.2662	0.89968	−0.5502	0.7498	0.84767	−0.5566	0.3086	0.89711	−0.6027	0.7990	0.83787	−0.4930
0.3663	0.89313	−0.6493	0.8501	0.82598	−0.4032	0.4028	0.89041	−0.6800	0.8995	0.81255	−0.3035
0.4611	0.88548	−0.7029	0.9495	0.79635	−0.1744	0.5118	0.88060	−0.7052			

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x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^{E}_{exp} 1)	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^{E}_{exp}	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^{E}_{exp}	x	$\frac{\rho}{(g\cdot cm^{-3})}$	V^{E}_{exp}
$(1-x)$(1,8-cineole) + $x(CH_3(CH_2)_2OH)$, 298.15 K											
0.0610	0.91673	−0.1920	0.5528	0.88249	−0.7235	0.1133	0.91529	−0.3202	0.6016	0.87687	−0.7075
0.1545	0.91324	−0.4043	0.6540	0.87017	−0.6725	0.2025	0.91063	−0.4885	0.6997	0.86378	−0.6380
0.2581	0.90733	−0.5749	0.7505	0.85585	−0.5795	0.3288	0.90256	−0.6533	0.8002	0.84717	−0.5047
0.3511	0.90090	−0.6706	0.8529	0.83682	−0.4087	0.4070	0.89650	−0.7083	0.8987	0.82671	−0.3072
0.4504	0.89276	−0.7297	0.9517	0.81348	−0.1688	0.5046	0.88760	−0.7370			
$(1-x)$(1,8-cineole) + $x(CH_3(CH_2)_2OH)$, 308.15 K											
0.0600	0.90899	−0.1732	0.5022	0.87923	−0.7295	0.1003	0.90721	−0.2745	0.5586	0.87331	−0.7232
0.1460	0.90501	−0.3758	0.6053	0.86795	−0.7118	0.2050	0.90184	−0.4846	0.6525	0.86198	−0.6879
0.2499	0.89915	−0.5488	0.7017	0.85504	−0.6402	0.3215	0.89437	−0.6287	0.7464	0.84811	−0.5885
0.3548	0.89195	−0.6597	0.7987	0.83909	−0.5135	0.4029	0.88819	−0.6975	0.8398	0.83118	−0.4367
0.4511	0.88404	−0.7178	0.8931	0.81975	−0.3240						
$(1-x)$(1,8-cineole) + $x((CH_3)_2CHOH)$, 298.15 K											
0.0976	0.91487	−0.2606	0.5740	0.87094	−0.5298	0.1444	0.91187	−0.3254	0.6162	0.86506	−0.5310
0.1929	0.90851	−0.3818	0.6594	0.85859	−0.5121	0.2415	0.90491	−0.4339	0.7161	0.84930	−0.4793
0.2942	0.90065	−0.4754	0.7689	0.83956	−0.4264	0.3472	0.89601	−0.5091	0.8045	0.83247	−0.3920
0.3879	0.89214	−0.5236	0.8484	0.82287	−0.3316	0.4482	0.88597	−0.5422	0.8949	0.81159	−0.2535
0.5052	0.87953	−0.5489	0.9493	0.79678	−0.1437						
$(1-x)$(1,8-cineole) + $x((CH_3)_2CHOH)$, 308.15 K											
0.0529	0.90892	−0.1875	0.5436	0.86603	−0.5369	0.1491	0.90294	−0.3350	0.5980	0.85885	−0.5303
0.2258	0.89739	−0.4093	0.6432	0.85229	−0.5141	0.2580	0.89490	−0.4420	0.6986	0.84346	−0.4810
0.2999	0.89144	−0.4706	0.7547	0.83357	−0.4452	0.3555	0.88649	−0.5034	0.8021	0.82427	−0.3994
0.3982	0.88244	−0.5294	0.8441	0.81522	−0.3487	0.4333	0.87881	−0.5349	0.8926	0.80357	−0.2677
0.4931	0.87220	−0.5431	0.9418	0.79027	−0.1613						
$(1-x)$(1,8-cineole) + $x(CH_3(CH_2)_3OH)$, 298.15 K											
0.0453	0.91789	−0.1503	0.5025	0.88375	−0.6607	0.1157	0.91411	−0.3144	0.6001	0.87297	−0.6450
0.1576	0.91162	−0.3935	0.6502	0.86680	−0.6195	0.2066	0.90845	−0.4649	0.6991	0.86025	−0.5771
0.2571	0.90498	−0.5336	0.7467	0.85340	−0.5269	0.3014	0.90169	−0.5788	0.7995	0.84516	−0.4582
0.3886	0.89455	−0.6402	0.8471	0.83705	−0.3792	0.4096	0.89269	−0.6490	0.8973	0.82774	−0.2793
0.4645	0.88756	−0.6646	0.9455	0.81797	−0.1650						
$(1-x)$(1,8-cineole) + $x(CH_3(CH_2)_3OH)$, 308.15 K											
0.1158	0.90551	−0.3082	0.5499	0.87040	−0.6625	0.1624	0.90273	−0.3935	0.6016	0.86453	−0.6465
0.2103	0.89965	−0.4665	0.6479	0.85887	−0.6228	0.2619	0.89609	−0.5324	0.7044	0.85137	−0.5760
0.3244	0.89138	−0.5924	0.7491	0.84497	−0.5313	0.3705	0.88767	−0.6317	0.7958	0.83773	−0.4676
0.4063	0.88457	−0.6492	0.8466	0.82917	−0.3832	0.4494	0.88061	−0.6608	0.8975	0.81979	−0.2788
0.5030	0.87536	−0.6685	0.9451	0.81028	−0.1660						
$(1-x)$(1,8-cineole) + $x((CH_3)_2CHCH_2OH)$, 298.15 K											
0.0523	0.91737	−0.1678	0.5544	0.87551	−0.7686	0.1135	0.91380	−0.3206	0.6029	0.86957	−0.7566
0.1744	0.90986	−0.4431	0.6684	0.86082	−0.7191	0.2167	0.90690	−0.5159	0.7092	0.85487	−0.6792
0.2629	0.90348	−0.5885	0.7498	0.84854	−0.6292	0.3050	0.90014	−0.6426	0.7966	0.84070	−0.5561
0.3690	0.89464	−0.7051	0.8502	0.83092	−0.4533	0.4053	0.89128	−0.7297	0.8980	0.82137	−0.3370
0.4534	0.88658	−0.7561	0.9426	0.81168	−0.2100	0.5038	0.88127	−0.7705			
$(1-x)$(1,8-cineole) + $x((CH_3)_2CHCH_2OH)$, 308.15 K											
0.0585	0.90845	−0.1865	0.5537	0.86715	−0.7641	0.1079	0.90553	−0.3015	0.6044	0.86098	−0.7502
0.1650	0.90189	−0.4196	0.6509	0.85482	−0.7176	0.2075	0.89896	−0.4942	0.7053	0.84711	−0.6738
0.2584	0.89521	−0.5711	0.7486	0.84048	−0.6265	0.3112	0.89103	−0.6374	0.7971	0.83242	−0.5525
0.3554	0.88730	−0.6860	0.8465	0.82350	−0.4582	0.4018	0.88307	−0.7186	0.8970	0.81354	−0.3391
0.4503	0.87838	−0.7482	0.9437	0.80347	−0.2050	0.5088	0.87225	−0.7649			

1) 单位为 $cm^3\cdot mol^{-1}$

表 3　各体系的拟合参数 A_i 和标准偏差 $\sigma/(cm^3\cdot mol^{-1})$

Tab.3　The least-squares parameters A_i and standard deviations $\sigma/(cm^3\cdot mol^{-1})$

1,8-cineole +	A_0	A_1	A_2	A_3	A_4	A_5	σ
甲醇(298.15)	−3.27116	−0.33162	−0.37417	0.92836	−0.69699	−1.59785	0.0043
甲醇(308.15)	−3.34491	−0.10704	−0.22797	0.15736	−0.85347	−0.69291	0.0040
乙醇(298.15)	−2.76827	−0.01234	−0.20392	0.32892	−0.51580	−0.32270	0.0030
乙醇(308.15)	−2.82624	0.01910	−0.00685	0.65941	−0.96197	−0.64179	0.0031
异丙醇(298.15)	−2.20632	0.08559	−0.42776	−0.09047	−0.82059	−0.30993	0.0034
异丙醇(308.15)	−2.17853	−0.03474	−0.33150	0.96348	−1.35194	−1.81055	0.0031
正丙醇(298.15)	−2.93676	0.04672	−0.28254	0.12219	−0.47225		0.0029
正丙醇(308.15)	−2.91380	0.19916	−0.42244	0.00326	−0.07995		0.0026
正丁醇(298.15)	−2.67229	0.02620	−0.35799	−0.12654	−0.47769		0.0023
正丁醇(308.15)	−2.67543	0.05464	−0.40624	−0.05139	−0.26685		0.0022
异丁醇(298.15)	−3.08012	0.32620	−0.36345	−0.03259	−0.35423		0.0019
异丁醇(308.15)	−3.05228	0.33450	−0.24511	−0.01534	−0.55859		0.0026

2 讨　论

(1) 从图 1 可知，桉叶油素与醇类 6 个体系的过量体积在全浓度范围内均为负值，在摩尔组成 $x=0.5$ 附近达到最小值，说明桉叶油素与醇类形成分子间络合物，引起体积显著收缩．对同一体系的过量体积来说，温度影响较小，308.15 K 的 V^E 略微小于 298.15 K 的 V^E．

(2) 对于液体混合物过量体积 V^E 的贡献主要来自物理化学作用和结构作用的结果．桉叶油素与醇类二元混合物 V^E 均为负值，可能是桉叶油素分子中的氧原子与醇分子中羟基的氢原子形成氢键络合物，是弱化学作用，对 V^E 是负值的贡献；另方面，在桉叶油素存在时，混合物中的物理作用和醇聚体解缔产生对 V^E 正值的贡献，总得说来负值的贡献是主要的．对于直链烷基醇其聚体随着烷基醇链长的增加而减少，而且高碳烷基醇给出质子能力比低碳烷基醇低．因此，桉叶油素与醇类各二元混合物中形成氢键络合物的作用随烷基醇链长的增加而降低[12]．对于同一个 x，V^E 随着醇分子碳链的增长而增加，依次为 CH_3OH < $CH_3(CH_2)_2OH$ < C_2H_5OH < $CH_3(CH_2)_3OH$．此外，桉叶油素与异构醇类的 V^E 通常大于相应正构醇类 V^E 值，如 $(CH_3)_2CHOH$ > $CH_3(CH_2)_2OH$[13]，主要原因是异构醇即支链烷基醇的空间位阻增加，难于形成氢键络合物的缘故．

桉叶油素与醇类这 6 个二元混合物的过量体积，$x=0.5$ 时，V^E 大小顺序为：CH_3OH < $(CH_3)_2CHCH_2OH$ < $CH_3(CH_2)_2OH$ < C_2H_5OH < $CH_3(CH_2)_3OH$ < $(CH_3)_2CHOH$．

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Excess Volumes of {1, 8-cineole + alkanols} at 298.15 and 308.15 K

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Abstract: The excess molar volumes V^E of {1, 8-cineole + CH_3OH, + C_2H_5OH, + $CH_3(CH_2)_2OH$, + $(CH_3)_2CHOH$, + $CH_3(CH_2)_3OH$, + $(CH_3)_2CHCH_2OH$} were determined from density measurements using a vibrating-tube densimeter at 298.15 and 308.15 K. All the mixtures studied exhibit negative excess molar volumes. The V^E values are fitted to a Redlich-Kister equation.

Keywords: 1, 8-cineole; excess molar volumes; vibrating-tube densimeter

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Metabolites of Marine Fungus *Hypoxylon oceanicum* from the South China Sea

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Abstract: A new sesquiterpenoid lactone 8-hydroxy-9-one-7(11)-eremophilien-12,8-olide(**A**), together with seven known compounds **C~H**, were isolated from the metabolites of marine fungus Hypoxylon oceanicum (326#) from the South China Sea. Their structures were established by using a broad range of spectral evidence including 2DNMR spectroscopic experiment.

Keywords: marine fungus; *Hypoxylon oceanicum*; metabolite; 8-hydroxy-9-one-7(11)-eremophilien-12,8-olide