Full Papers

微量热法测定RE(Et₂dtc)₃(phen)配合物的比热容

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摘要 推导了用改进的RD496-III型微热量计测定固态物质比热容的计算式. 用Joule

效应确定了仪器在298.15 K时的量热常数和精度分别为63.901±0.030

μV/mW和0.3%, 用Peltier效应测定总不平衡热.

在该仪器上测定的两种标准物质(基准苯甲酸和 α -Al $_2$ O $_3$)比热容的计算值与文献值相差在0.4%以内. 用本法测定了13种固态配合物RE(Et $_2$ dtc) $_3$ (phen) (RE = La, Pr, Nd, Sm~Lu)的比热容值,

与稀土原子序数 Z_{RE} 作图呈现三分组现象,说明配合物中 RE^{3+} 与配体间的化学键有一定程度的共价性,显示了稀土离子4f电子云的扩大效应.

关键词 <u>微量热法,不平衡热,固态配合物,比热容</u> 分类号

Determination of the Specific Heat Capacity of RE(Et₂dtc)₃(phen) by Microcalorimetry

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Abstract A calculation formula for determining the specific heat capacity of solid compound with an improved RD496-III microcalorimeter was derived. The calorimetric constant and precision determined by the Joule effect were $(63.901\pm0.030)~\mu\text{V/mW}$ and 0.3% at 298.15 K, respectively, and the total disequilibrium heat has been measured by the Peltier effect. The specific heat capacities of two standard substances (benchmark benzoic acid and α -Al $_2$ O $_3$) were obtained with this microcalorimeter, and the differences between their calculated values and literature values were less than 0.4%. Similarly, the specific heat capacities of thirteen solid complexes, RE(Et $_2$ dtc) $_3$ (phen) (RE=La, Pr, Nd, Sm—Lu, Et $_2$ dtc: diethyldithiocarbamate ion, phen:1,10-phenanthroline) were gained, and their total deviations were within 1.0%. These values were plotted against the atomic numbers of rare-earth, which presents tripartite effect, suggesting a certain amount of covalent character in the bond of RE $^{3+}$ and ligands, according to Nephelauxetic effect of 4f electrons of rare earth ions.

Key words microcalorimetry disequilibrium heat solid complex specific heat capacity

DOI:

扩展功能

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