



SDI Review Form 1.6

Journal Name:	American Chemical Science Journal
Manuscript Number:	Ms_ACSJ_28258
Title of the Manuscript:	Thermal Properties, Crystal Structure, and Phase Transition of Racemic CaC₄H₄O₆•4H₂O Single Crystals
Type of the Article	Original Research Article

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This journal's peer review policy states that **NO** manuscript should be rejected only on the basis of '**lack of Novelty**', provided the manuscript is scientifically robust and technically sound.

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PART 1: Review Comments

	Reviewer's comment	Author's comment (if agreed with reviewer, correct the manuscript and highlight that part in the manuscript. It is mandatory that authors should write his/her feedback here)
Compulsory REVISION comments	The TGA pathway must be incorrect. If calcium oxalate is an intermediate, then it decomposes < 500 deg C under loss of CO to produce CaCO₃ as it was shown by several independent laboratories. E.g. see: Weight Loss Determined from Mass Spectrometry Trend Data in a Thermogravimetric/Mass Spectrometer System Carlton G. Slough TA Instruments, 109 Lukens Drive, New Castle DE 19720, USA That means, that the proposed thermal decomposition pathway must be incorrect as the decomposition of CaC₂O₄ was supposed to occur at >700 deg C under loss of oxygen. I think it likely to be a transition from the water free tartrate to the ethendiolate salt under loss of CO (not H₂CO) and then followed by a loss of H₂O not (1/2 O₂). I know this was proposed	We wish to thank you for your informative comments. We understand the reviewer's comment regarding the thermal decomposition of CaC₂O₄. It has been reported that the endothermic peaks at around 790 and 1040 K in the DTA curve of CaC₂O₄·H₂O are produced in nitrogen atmosphere by the elimination of CO and CO₂ gases. However, as described in this manuscript, small exothermic and endothermic peaks at around 750 K are found in the DTA curve under N₂ gas flow. We believe that the formation and elimination of gas occur at this temperature. Moreover, according to the reviewer's comment, it is assumed that the weight losses of CaC₂O₄ due to the decomposition are driven by the elimination of CO and CO₂ gases. Then, the theoretical weight losses are calculated to be 10.8% and 16.9%, respectively. These values are different from the experimental weight loss values of 8.2% and 22.9%, respectively. The theoretical weight loss values of 6.1% and 21.5% calculated from the elimination of (1/2)O₂ and 2CO gases in the manuscript are close to the experimental values, respectively. Thus, we do not accept the suggestion of the TGA pathway. According to the reviewer's suggestion, the chemical reactions involved in the thermal decomposition are described by the following chemical equations: $\text{CaC}_4\text{H}_4\text{O}_6 \rightarrow \text{CaC}_2\text{H}_4\text{O}_4 + 2\text{CO}$ $\text{CaC}_2\text{H}_4\text{O}_4 \rightarrow \text{CaC}_2\text{O}_2 + 2\text{H}_2\text{O}$ $\text{CaC}_2\text{O}_2 \rightarrow \text{CaC} + \text{CO}_2.$ The remaining substance is CaC, as suggested by the chemical equations described above. The reviewer's suggestion does not yield the result, which the substance is



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	in ref 14 but the numbers in terms of quantitative weight loss make much more sense with this pathway than the proposed one – in both instances. If not exactly in the same detail, this work including the crystallization method has been published before and the authors have not referenced this work: International Journal of Mathematics and Physical Sciences Research ISSN 2348-5736 (Online) Vol. 3, Issue 1, pp: (9-15), Month: April 2015 - September 2015, Available at: www.researchpublish.com Page 9 Research Publish Journals Structural, Optical and Thermal Characterization of Calcium Tartrate Crystal Grown Under the Influence of Magnetic Field R.G. VIDHYA1, R. RAMASAMY2, L. VIJAYALAKSHMI3 The question is why and why did the Thermal Analyses look so different with such different decomposition pathways? The missing discussion of such deviating results is concerning	known to be CaO. The paper written by Vidhya et al. surely contains some errors. The chemical reactions involved in the decomposition are described by the following chemical equations: $\text{CaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O} \rightarrow \text{CaC}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{O}$ $\text{CaC}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O} \rightarrow \text{CaC}_4\text{O}_4 + 2\text{H}_2\text{O} + \text{H}_2\text{O}$ $\text{CaC}_4\text{O}_4 \rightarrow \text{CaCO}_3 + \text{C}_3\text{O}$ The evaporated substance at last is C₃O, as suggested by the equations described above. The compound with formula C₃O does not exist in nature. The calculated values of the theoretical weight losses at each temperature range are incorrect. It is found that they have neglected a small weight loss at around 720 K in the TG curve, etc. From the facts and results mentioned above, the chemical equations for the thermal decomposition of DL-CaC₄H₄O₆·4H₂O and the conclusion in this paper have not been changed.
Minor REVISION comments		
Optional/General comments	Otherwise it is a well-written document with few errors.	Thank you