Modern techniques create demand for new monocrystalline materials having specific properties, including metal selenites. This work is devoted to the development of zirconium diselenite monocrystal synthesis by use of thermophysical processes. Zirconium will form only an amorphous basic aqueous selenite of indefinite structure and waterless normal zirconium selenite – diselenite Zr(SeO₃)₂. Zirconium diselenite has a crystalline constitution and, therefore, is the single reliably identified selenite of zirconium. Zirconium diselenite decomposes at the temperature of 873 K on dioxide zirconium and selenium dioxide. This makes it impossible to grow zirconium diselenite monocrystals from a melt. In this investigation it has been established that zirconium diselenite dissolves completely at 673 K and specific molar ratio Zr(SeO₃)₂ and SeO₂ in the liquid selenium dioxide existing at this temperature at a pressure approximately of 1.22 MPa. Development of a method and investigation of preparing single crystal process were carried out by dissolution of zirconium diselenite in liquid selenium dioxide in specially constructed ampules and heating with subsequent transpiration of the dissolvent. During removal and the subsequent condensation of selenium dioxide vapours there was a selection of zirconium diselenite in the form of single crystals. Single crystals of zirconium diselenite are colourless. Refraction indices were determined by means of a standard immersion set and were appeared more than 1.780 – 98. The conoscopic figure for all crystals is a cross with centre in sight, which disintegrating on a hyperbola at crystal twirl. The crystals of this type concern to biaxial crystals of monoclinic singonia with the main cleavage on a pinacoid or a prism according to the table for definition of cleavage, axality and singonia mineral forms. With the assistance of chemical analysis, X-raying, derivatography and infrared spectroscopy it is established that single crystals prepared based on thermophysical processes have a structure corresponding to the formula Zr(SeO₃)₂.