A process and arrangement for separating and recovering polyol production wastes, including polyol, potassium or sodium phosphate and magnesium silicates, is disclosed. According to the invention, the polyol product waste and caustic salt are combined in a vessel, emulsified and then deemulsified. This causes the contents of the vessel to stratify into three layers. A first stream containing primarily magnesium silicates and a minor amount of caustic salt solution is withdrawn from the vessel and then water-washed. The water-washed magnesium silicates are then dehydrated and desiccated to obtain commercial grade magnesium silicates. The minor amount of caustic salt solution is dehydrated, crystallized and then desiccated to yield commercial grade anhydrous potassium or sodium phosphate. A second stream containing predominantly polyol oil is withdrawn from the vessel and subsequently filtered and dehydrated to obtain commercial grade polyol. The aforementioned process steps can be accomplished by an arrangement of commercially available equipment.