

AD/A-005 860

CRYSTALLIZATION OF LEAD AZIDE

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Charlottesville, Virginia**

4 September 1973

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In Reply Refer to:
FSTC-HT-23-901-73 ✓
DIA Task No. T70-23-01

Date: 4 September 1973

ENGLISH TITLE: Crystallization of Lead Azide

SOURCE: Izv. Tomsk. Politekh. Inst., Vol. 199, 1969, pp. 64-65.

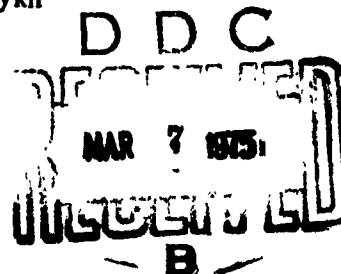
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LANGUAGE: Russian

COUNTRY: USSR

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Among the works on the thermal, photo- and radiation decomposition of heavy metal azides, only a very limited number of studies has been performed using large single crystals. However, it is obvious that only studies on perfect crystals can produce correct results on the kinetics and geometry of the decomposition reactions in solid crystalline compounds with controllable values of surface area. Among the works available to us involving solid solutions in the systems of heavy metal azides, we found reports on the production of large and defect-free single crystals, with perfect structure and sufficiently large for the study of the electro-physical properties and concentrations of photolytic and radiolytic products, by methods described in [1, 2].

Unfortunately, no detailed description of the methods of preparation of PbN_6 crystals could be found in the literature. Only [2, 3] provided the most general indications of the methods to be used for the preparation of PbN_6 crystals.

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AD A 005860

This work describes the method for preparation of PbN_6 crystals from solution in a saturated solution of ammonium acetate by slow cooling, growing crystals both of pure PbN_6 and with homophase additives of Ag^+ and Cu^{++} .

Materials and Apparatus

The initial substance used was polycrystalline PbN_6 , prepared by coprecipitation upon decantation of 0.2 N solutions of technical, twice recrystallized NaN_3 and $\text{Pb}(\text{NO}_3)_2$ of "chemically pure" grade. To introduce the homophase Ag^+ and Cu^{++} additives, the corresponding quantity of AgNO_3 or $\text{Cu}(\text{NO}_3)_2$ was added to the initial $\text{Pb}(\text{NO}_3)_2$ solution. Ammonium acetate of "chemically pure" ^{classification} was used to dissolve the PbN_6 .

The crystals were grown in a type U-10 thermostat; the required cooling rate of the solutions was maintained automatically using a clockwork mechanism connected to the magnetic head of a contact thermometer by a gear system.

The monocrystals were grown in a 100 ml chemical beaker in U-shaped tubes.

Experiments

The solvent used was a 17% solution of ammonium acetate in water. This solution was saturated with PbN_6 at 70-90° C for 2-3 hr. The excess PbN_6 was then filtered off in a hot filter funnel, and the solution was poured into the vessels in which crystallization was conducted.

In growing our PbN_6 single crystals, we encountered the difficulty of preventing spontaneous explosions as described in [3, 4]. It was established that the probability of a spontaneous explosion, as well as the temperature at which it occurs, depend on the quantity of solution in the crystallizer and the cooling rate. With the identical cooling rate of 2°/hr, spontaneous explosions in 100 ml of PbN_6 solution occur in the 35-45° C temperature interval, while in 25 ml of this same solution they occur at 15-20° C.

As the cooling rate increases, the probability of a spontaneous explosion upon passage through the critical temperature increases.

The crystallization of PbN_6 usually occurs in the 20-25° C temperature interval. The addition of Ag^+ increases this temperature to 40-42° C, while the addition of Cu^{++} decreases the crystallization temperature to 17-19° C.

Considering the above, the following method was selected for growing of PbN_6 crystals, allowing the probability of spontaneous explosion to be minimized.

The solution was cooled from the temperature at which it was saturated with PbN_6 to a temperature $1-2^\circ$ higher than the crystallization temperature at a rate of $2^\circ/\text{hr}$. The cooling rate was then reduced to 1° per 12-14 hr. It is preferable to work with quantities of solution of not over 25 ml. In the U-shaped tubes, the crystals grow quite large. Crystallization under these conditions succeeded in completely eliminating explosions.

The PbN_6 crystals produced are white and nontransparent. Upon long storage in darkness, they become pale pink. The addition of Ag^+ does not influence the color of the crystals, while the addition of Cu^{++} causes the crystals to become reddish brown.

The PbN_6 crystals usually had the form of an irregular, extended hexagon, reaching $6 \times 1 \times 1$ mm or (less frequently) a parallelepiped with maximum dimensions of $5 \times 3 \times 1$ mm. The PbN_6 crystals reached 4-5 mg in weight.

The addition of Ag^+ increases the dimensions of the crystals (crystals measuring $11 \times 2 \times 2$ mm and weighing 7-8 mg were produced). Crystals with Cu^{++} were not grown larger than $3 \times 2 \times 0.5$ mm.

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