

TABLE I
DATA FOR LANTHANUM HYDROXIDE SOLS

Sol	NH ₄ OH, % of equiv.	Hours dialyzed ^a	La ₂ O ₃ , mg. per l.	Cl, mg. per l.	pH
6c	33.3	40	250	22.9	6.9
6d	33.3	45	500	17.2	7.0
6e	33.3	48	829	34.4	7.4
6f	33.3	51	755	37.0	
6g		84	438	10.5	7.3
6z	33.3	120	553	15.0	7.0
6h(1)	50.0	67	1158	103	7.8
6h(2)	67.0	67	1984	60.7	8.3
6h(3)	89.0	67	2312	48.7	7.9

^a All sols dialyzed at 20 to 25°.

chloride is necessary for stability. When protected from carbon dioxide, the more dilute sols are reasonably stable, but slow sedimentation takes place in the more concentrated ones. The sols are bluish-white by both reflected and transmitted light, but on strong illumination, the light transmitted is rich in the orange, a qualitative verification of the Rayleigh equation. The particles are positively charged.

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Preparation of Pellets of Radioactive Lead

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In the lead mirror method of detecting free radicals,² the lead used must be as pure as possible; the presence of oxide is distinctly objectionable. In experiments in which the lead transported is measured by the use of a radioactive isotope (Ra D) it is found necessary to prepare lead pellets of minimum size and maximum purity so as to ensure maximum sensitivity of the method. Various methods of preparation, which might be expected to work but generally have proved unsatisfactory, are described in the literature. We have found that the one described below, based upon a reaction used by Stas,³ gave pellets of desirable form and character.

Several hundred crushed radon tubes were triturated in a Pyrex mortar with concentrated aqua regia for a prolonged period of time, and the extract filtered, through paper, into a quartz beaker. The treatment with concentrated acids was re-

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(2) Cf. Rice and Rice, "The Aliphatic Free Radicals," The Johns Hopkins Press, Baltimore, Md., 1935.

(3) Stas, *Bull. Acad. Belg.*, [2] 10, 295 (1860); *Chem. News*, 4, 307 (1861).

peated on the siliceous residue, and the two filtrates combined, giving a total volume of 60-70 cc. At this point a definite weight of pure lead nitrate was added, the quantity depending on the size of pellet and the degree of radioactivity desired; in the following procedure the amounts stated are for the preparation of a 0.1-g. pellet of lead.

The next step was the removal of nitric acid by repeated evaporation to dryness with hydrochloric acid, on a hot plate. The residue was then dissolved in 33 cc. of 5 *N* hydrochloric acid, and treated with hydrogen sulfide to precipitate the mercury (contained in the radon tubes as a result of the method of filling the latter), the mercuric sulfide being filtered off on paper and washed with hot dilute hydrochloric acid. The filtrate so obtained was again evaporated to dryness, repeatedly, this time with water, to remove hydrochloric acid, and the residue of lead chloride crystals was dissolved by warming in 30 cc. of water. The removal of the hydrochloric acid is necessary in order to precipitate the lead as oxalate from a neutral solution without too high a concentration of ammonium salts resulting from the neutralization of acid. To the cold aqueous solution, then, was added 4 cc. of saturated ammonium oxalate solution and 2-3 drops of dilute ammonium hydroxide. After stirring and thorough cooling the mixture was allowed to stand overnight. The precipitate was then filtered on a Neubauer porcelain microcrucible, with suction, washed with cold water, and dried partially with air. This was followed by thorough drying (and partial decomposition to oxide) at 250°.

For the reduction to the metallic state, the dry residue of oxalate + oxide was transferred into an ordinary porcelain crucible, and covered with 1-2 g. of dry potassium cyanide. The inclined, open crucible was heated slowly and carefully on a Bunsen flame, and finally heated very strongly for about ten minutes. On cooling, the potassium cyanide was washed away with water, and the lead pellet picked out and dried. If the lead thus finally obtained should prove to be powdery, or finely divided rather than in the form of one or a few pellets, the potassium cyanide treatment is repeated on the dry lead, until the metal is obtained in a satisfactory pellet or two.

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