

# LABORATORY MANUFACTURE OF DRY LEAD SUBACETATE

By G. S. MOBERLY.

During the 1943/44 season supplies of dry lead acetate became exhausted, and it became necessary to make our own stocks.

The procedure adopted was based on verbal information given to the writer by R. M. Bechard. This was modified as the result of experiment.

If the appropriate quantities of neutral lead acetate and litharge are ground together in a mortar, the mixture after a few minutes turns to a heavy pink paste. With further grinding this becomes heavier and assumes the consistency of nearly dry putty. On still further grinding the pink colour begins to fade and the heavy paste crumbles to a white powder. For small quantities in a laboratory mortar the whole process takes a little over an hour. With larger quantities the grinding naturally takes longer.

At first, quantities were taken in the proportions used for making lead subacetate solutions, viz., 43 grams neutral lead acetate to 13 grams of litharge. Analysis of the product showed a deficiency of basic lead. Eventually the litharge was increased to 17 grams to 43 grams of neutral lead acetate. This gave a product closely approximating to the theoretical formula of four parts of  $3\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot \text{PbO}$  and three parts of  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{PbO}$  as given by Browne.<sup>1</sup>

In the manufacture of larger quantities the following procedure was eventually devised.

In a large iron mortar with a glazed surface, 1720 grams of neutral lead acetate and 680 grams of litharge were ground with a large earthenware pestle. After about two hours grinding the mixture, which by this time had passed its most sticky stage and had begun to get slightly crumbly, was placed on a large enamelled tray and rolled like pastry with a glass rolling-pin. It was then spread out over the tray and placed in the sun for an hour. After that it was placed in a steam-heated oven and heated below  $100^\circ\text{C}$ . for one hour. If the temperature was allowed to rise over  $100^\circ$  the material caked and became difficult to grind. It was then returned to the mortar and ground until it crumbled to a powder.

Now the powder was sifted through a 100-mesh sieve. The retained particles were returned to the mortar, reground and re-sifted. This procedure was repeated three or four times. A further spell in the drying oven at this stage would have been advantageous, but unfortunately the oven space at our disposal was very limited.

Only about 70 per cent. of the original weight was finally obtained in the form of a fine powder. The balance consisted of hard grains, about 1 mm. in diameter, which resisted all attempts at grinding with the means at our disposal. This residue was not analysed, but probably consisted of carbonate formed by contact with the  $\text{CO}_2$  of the atmosphere.

No difference could be found when using either yellow lead oxide or red lead. The use of a plain iron mortar and pestle proved unsatisfactory, as a considerable quantity of iron appeared in the final product. One curious phenomenon, so far unexplained, was that during experimental grindings on one or two occasions the pasty stage never appeared at all, and the mixture remained powdery at all stages.

An analysis of the final product was:—

	Sample.	Theoretical.
Moisture ... ..	3.1 per cent.	—
Total lead dry basis ... ..	71.6 per cent.	72.84 per cent.
Basic lead dry basis ... ..	28.9 per cent.	29.14 per cent.
Basic per cent. total lead ...	40.4 per cent.	40.00 per cent.

I am indebted for help and advice to Mr. D. Elysée, who was doing similar work on his own account at Amatikulu, and to Mr. J. Hellet, who supervised the actual manufacture; also to Mr. J. L. du Toit, of the Experiment Station, for chemical analyses of numerous samples.

## Reference.

<sup>1</sup> Brown, C. A. (1912): Handbook of Sugar Analysis, 214.

Mr. MOBERLY said that before he undertook to make dry lead subacetate in the laboratory, he approached certain firms in Durban to see whether they would be prepared to take it up. They, however, declared their inability to do so. Machinery which was impossible to obtain as a result of the war was required, and also there was only a relatively small demand in this country. In addition the nature of the product made the selection of a grinder very difficult, as a powder of uniform fineness was required.

He had conducted many comparative polarization tests on juices, using his own product and dry lead subacetate sold by reputable firms for clarification, and only in one case was there a difference, and that was not a fair comparison as the sample of lead subacetate was old and probably deteriorated.

Mr. RAULT pointed out that in handling dry lead subacetate powder in bulk there was the danger of breathing in the dust and contracting lead poisoning. He had had this happen to one of his boys, who was sick for about ten days.

Mr. MOBERLY said he tried to guard against this danger in the grinding process, but usually there was very little danger as moist conditions prevailed to the final stage, which came suddenly. In one case, however, grinding was carried too far and the powder became completely air-borne and quite impossible to handle.