

The contents of the tubes were worked up as already outlined. Succinimide was found in all of the tubes, the amount increasing with the rise in temperature and the duration of the heating. No. 1 contained much unchanged cyanide. Small amounts of amic acid occurred in all of these tubes, and traces of what appeared to be amide were separated in several.

No. 5 contained 5 grams of ethylene cyanide, two molecules of water, and no sulphuric or other mineral acid. It was heated for five hours at 153° to 173° . The mixture in the tube then appeared as a brownish liquid containing oily globules, and with a strong odor of ammonia. Most of the cyanide appeared unchanged, as scarcely any imide could be detected.

No. 6 carried 5 grams of the cyanide, one molecule of water, and no mineral acid. It was heated for six hours at 153° to 173° . The tube contents were then brown and semisolid. Succinimide in large amount was separated, a small amount of amic acid, traces of succinamide, but no cyanpropionamide was discovered.

For Glutarimide.—Trimethylene cyanide was heated in sealed tubes with two molecules of water and half a molecule of sulphuric acid, for five to ten hours at 155° to 200° , and the products of the reaction separated in essentially the same manner as for the succinimide tubes. Glutarimide was isolated in large amount (60 per cent. of theory), but no amide or amic acid could be detected, except the imide, the tubes containing only unhydrated cyanide. A temperature of 180° to 200° is necessary for the success of the hydration.

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ON THE DETERMINATION OF CITRATE-INSOLUBLE PHOSPHORIC ACID.

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THE method used in the laboratory of the North Carolina Department of Agriculture up to recently was as follows:

Two grams of the sample to be analyzed were washed free of water-soluble phosphoric acid. Then 100 cc. of strictly neutral ammonium citrate solution (sp. gr. 1.09) in an 8-ounce Erlenmeyer flask, was heated to 65° , in a water-bath, keeping the

flask loosely stoppered to prevent evaporation. When the citrate solution in the flask had reached 65° , the filter containing the washed residue from the water-soluble phosphoric acid determination was dropped in and the flask stoppered tightly and shaken until the filter-paper was reduced to a pulp. The flask was then placed back, loosely stoppered, in the water-bath, the temperature of which was so maintained as to give exactly 65° in the citrate flask. The flask was shaken every five minutes. At exactly thirty minutes from the time the filter and the residue were introduced, the flask was removed from the bath and the contents filtered as rapidly as possible. The residue on the filter was then washed thoroughly with distilled water at 65° . The filter and its contents were then returned to the original digestion flask, and 40 cc. nitric acid and 20 cc. hydrochloric acid were added and boiled down to about 15 or 20 cc. concentration.

The solution was diluted to 200 cc., and 40 cc., corresponding to 0.4 gram of fertilizer, were measured into a 500 cc. Erlenmeyer flask. Add 10 or 12 grams ammonium nitrate and a little distilled water. The excess of acid is neutralized with ammonia. When the contents had cooled 30 cc. of recently filtered molybdic solution were added and the flask, after securely stoppering with an ordinary rubber stopper, was placed in a Wagner shaking machine which was revolved by a hot-air motor and shaken for thirty minutes. The shaking machine was maintained at from 45 to 55 revolutions per minute, as this velocity has been found to give the maximum agitating efficiency. The flask was removed from the shaking machine and contents filtered, washed, returned to the shaking flask, and titrated.

The method of filtering the contents of the flask, after heating with citrate solution, through a funnel in which was a very thick filter-paper and platinum cone and using pressure for rapid work, was found to be unsatisfactory in three ways:

First.—The filter-paper was likely to burst and thereby necessitate another filtration.

Second.—It was very difficult to fit the filter-paper in the funnel so that no air would get in around the sides and retard the filtration.

Third.—It was entirely too slow for rapid work.

Having encountered these difficulties, the Hirsch funnel was

tried, using a closely fitting filter-paper in the bottom, but it was found very difficult to fit this filter-paper so that none of the substance would pass down around the edges and render the determination valueless.

This method not proving satisfactory, the Hirsch funnel, using filter-paper pulp in the bottom, was next tried. It was found that in order not to allow any of the substance to pass through, it was necessary to use a large amount of pulp and this rendered the filtration as slow as in the first case.

This method being abandoned, a filter of the following construction was tried: Take a carbon filter, and place in the bottom of it a tightly fitting perforated porcelain disk, to which is attached a small wire that extends down beyond the small end of the carbon filter. A rubber stopper is then fitted tightly in a pressure bottle and the carbon filter passed through it. A layer of asbestos is placed on the disk in the carbon filter and by the aid of pressure this gave entire satisfaction.

By this method it took only from thirty to forty minutes to filter and wash the residue, while with the old way it took from one and a half to two hours.

Where a large number of samples are to be analyzed one will not only save a great deal of time but the residue can be washed more thoroughly.

Some difficulty was also experienced from the cracking of the ordinary rubber stopper used in the shaking flask, when revolving in the shaking machine, because the solution would leak out around the crack in the rubber stopper. To prevent this, antimony rubber stoppers were tried and these proved very successful as no leaking has been noticed since they were put in.

In another part of the method, rapidity will also be increased by proceeding as follows: Just before adding the molybdc solution, to precipitate the phosphoric acid, neutralize the excess of acid with ammonia, and then add 10 or 12 grams of ammonium nitrate; then the molybdc solution can be added immediately, for the ammonium nitrate cools the solution down to where it is safe to precipitate and no waiting to cool is necessary as is the case if ammonium nitrate is added before the excess of acid is neutralized.