

the conditions of preparation. The platinum salt examined under the microscope consisted of a mixture of crystals and amorphous masses.

QUATENARY NITRATE,  $C_{24}H_{28}N_3O.NO_3$ .

The salt is prepared by dissolving the quaternary chloride in hot water and adding a saturated solution of potassium nitrate in water. It is recrystallized from boiling water and dried in vacuo over sulphuric acid. Snow white rectangular prisms, difficultly soluble in cold water, quite soluble in methyl alcohol and hot water. It turns yellowish at  $190^\circ$  and melts at  $192-94^\circ$  to a reddish liquid.

Analysis gave: C, 65.60 per cent; H, 6.22 per cent.

Calculated for  $C_{24}H_{28}N_3O.NO_3$ ; C, 66.02; H, 6.47.

QUATENARY PICRATE,  $C_{24}H_{28}N_3O.C_6H_2N_3O$ .

The salt is prepared by dissolving 2 gm. of quaternary chloride in 500 cc. hot water and adding an excess of a hot solution of sodium picrate containing some free sodium carbonate. It is recrystallized from boiling water containing a little sodium carbonate and dried in vacuo. Soft, orange-yellow, oblong plates, difficultly soluble in all solvents. It turns reddish at  $145^\circ$  and melts at  $155^\circ$ .

It contained 13.96 per cent N.

Calculated for  $C_{24}H_{28}N_3O.C_6H_2N_3O$ , 13.96.

QUATENARY PICROLONATE,  $C_{24}H_{28}N_3O.C_{10}H_7N_3O$ .

The salt is prepared as follows: Sodium carbonate and picrolonic acid, one gm. each, are dissolved in 800 cc. warm water, and the solution set aside over night in a cool place. The liquid is filtered, and to the filtrate, heated nearly to boiling, is added a solution of one gm. quaternary chloride in 200 cc. hot water. The salt is recrystallized from boiling water, of which it requires about 2000 cc. for solution. Bright orange colored microscopic needles, very difficultly soluble in all solvents. Air dried it melts at  $164-66^\circ$  to thick liquid. For the estimation of N it was dried in vacuo.

The salt contained 15.86 per cent N.

Calculated for above formula, 15.38.

The investigation is to be continued.

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CALCIUM HYDROXIDE.

A Plea For Its Introduction Into the U. S. P.

PHILIP ASHER, PH. G.

The official method of making lime water is simple enough, and the principal question of interest is its only constituent, the lime, and which has caused an endless amount of unnecessary worry to those who have to make this solution.

The first difficulty the retailer meets is in obtaining lime of good quality.

Often lime is obtained which is difficult to slake, or, after having had it for a short time, one finds it has become air slaked and naturally it is thrown out.

The city druggist often experiences trouble in getting lime of good quality, and this difficulty is still greater in the country.

A common but highly erroneous practice is to slake lime in a demijohn, add water to it, and decant the clear solution when needed, adding water to the residue from time to time without limit.

Any intelligent pharmacist should know that a solution of calcium hydroxide absorbs carbon dioxide, forming the insoluble calcium carbonate. This notion that as long as there is a residue remaining it is calcium hydroxide is far more prevalent than one may believe and the writer has heard such a theory propounded from the lecture platform. While chairman of the Committee on Adulteration of the Louisiana State Pharmaceutical Association, 85 samples of lime water were examined, among which were some consisting of nothing but water, and strange as it may appear one of these was furnished by a member of the Board of Pharmacy.

It is seldom one meets with lime water containing an excess of alkali, but in the investigation above mentioned several such cases were observed, being due to improper washing.

Even were lime worth as much as 25 cents per pound and the full official amount were used a gallon of it would not cost half a cent, so that the charge of intentional cheapening could never be made against anyone. As an article of domestic consumption it is very important, and the pharmacist who supplies an inferior article is criminally negligent.

The writer undertook a series of investigations regarding the rate of the deterioration of lime and was surprised at the results.

That lime when exposed to the atmosphere absorbs carbon dioxide and water is correct, but the rate of such changes is not as rapid as generally believed. If a purified calcium hydroxide were introduced into the U. S. P. the pharmacist would have at his command the material with which to make lime water and without the necessity of washing.

On November 19, 1910, these experiments were begun :

"A"—Lime slaked, dried until it no longer lost weight, and placed in a 4 ounce, wide-mouth bottle, stoppered with an ordinary cork.

"B"—Same as "A," but kept in an open 4 ounce beaker and exposed to atmosphere.

"C"—Sufficient water was added to slake the lime and kept under same conditions as "A."

"D"—Same as "C," but kept exposed.

"E"—Lime placed in 4 ounce uncovered beaker and allowed to become air-slaked.

"F"—A preparation known as "lime opura," consisting of slaked lime. This was over six years old at the beginning of these experiments and was kept during that period in an ordinary cardboard box, and after the experiments were started was exposed to the atmosphere.

The following table shows the progressive changes.

Under "E" the reading is in terms of oxide instead of hydroxide.

It will be observed that the changes in the exposed samples during the first month were gradual, after which there was a decided drop, the latter occurring during the rainy spell, but since that time it has become almost stationary.

Date	A % Ca(OH) <sub>2</sub>	B % Ca(OH) <sub>2</sub>	C % Ca(OH) <sub>2</sub>	D % Ca(OH) <sub>2</sub>	E % CaO	F % Ca(OH) <sub>2</sub>
November 19, 1910.....	88.51		88.25			
November 21, 1910.....	87.5	84.09	86.6	83.		
November 24, 1910.....	86.8	78.	86.5	78.	78.88	77.5
December 3, 1910.....	86.5	76.5	86.5	70.25	65.98	76.9
December 10, 1910.....	86.45	75.4	86.5	70.15	65.95	76.8
December 17, 1910.....	86.43	74.29	86.44	69.8	65.95	75.4
January 1, 1911.....	83.4	28.75	86.44	37.44	52.61	38.37
February 11, 1911.....	83.35	28.47	86.44	35.30	46.76	37.42
March 4, 1911.....	83.29	27.95	86.43	33.10	45.65	37.25
May 4, 1911.....	83.22	20.96	86.43	32.53	45.58	37.08
July 11, 1911.....	83.12		86.43	30.89	41.20	36.41

The results under "A" and "C" are of the greatest interest to the pharmacist, showing that with no other precaution than to cork the bottle the changes in nearly eight months were from 88.51% to 83.12% in "A" and from 88.25% to 86.43% in "B," or a difference of 6.3% in the former and 2.5% in the latter.

The above results also disclose the fact that even slaked lime could be used, provided an increased amount has been taken and which could be shown to contain hydroxide by a drop of phenolphthalein solution.

A purified calcium hydroxide could be made by the average retail druggist, but the chemical and pharmaceutical houses are better equipped for such work and it could be marketed at a very reasonable figure.

#### DISCUSSION.

CHARLES H. LA WALL: "I have frequently observed that milk of lime does not deteriorate as rapidly as commonly supposed if kept under common sense conditions. I am glad that Dr. Asher has made the tests that he did."

F. R. ELDRÉD: "There have been many elaborate schemes proposed for the keeping of lime water, such as siphons and similar arrangements. Some time ago I made several experiments as to the rate of deterioration of calcium hydroxide in solution. One was the keeping of a gallon of lime water with an excess of calcium hydroxide, the bottle being stoppered with an ordinary cork. Once a week the bottle was uncorked and two ounces poured out, without shaking, until only about two ounces of solution remained above the lime. The liquid remained saturated all the time.

"Another gallon bottle of the solution was kept with simply a paper cover to exclude dust, and every week a portion was removed by means of a pipette and titrated. While the liquid did not remain absolutely saturated it was above the pharmacopœial requirement at all times."

#### IMPROVEMENT IN THE TECHNIQUE OF SAMPLING URINE FOR MICROSCOPIC EXAMINATION.

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Let it be assumed, for the purpose of illustration, that an adult male will void about 250 cc. of urine each time he empties his bladder; that the total volume of his urine in twenty-four (24) hours is about 1500 cc., and that the clinician will