

two showed a slight increase, two were unchanged, two showed a slight decrease, and two showed a marked decrease. It seems reasonable to suppose that adsorbed hydrogen ions would be driven off below 500° C., with a consequent increase in adsorptive capacity for methylene blue.

We are led to conclude that the observation that acid extraction does not in general lead to increased dye adsorption must be accounted for by the inaccessibility to methylene blue ions of the sites occupied by the basic compounds on the silica surface. The reduction in adsorptive capacity observed in many cases may arise from disruption of capillaries adjacent to the positions occupied by the basic compounds, or of capillaries of which these latter form structural units, leading to a general 'smoothing out' of the surface.

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¹ Lea, F. M., and Nurse, R. W., *J. Soc. Chem. Indust.*, 58, 277 (1939).

² Gaudin, A. M., and Rizo Patrón, A., *Trans. Amer. Inst. Mining Eng.*, 153, 462 (1943).

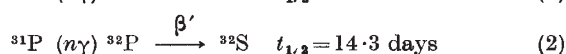
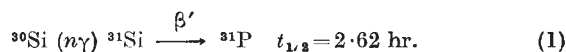
³ Gibb, J. G., and Ritchie, P. D., *J. App. Chem.*, 4, 473 and 483 (1954).

⁴ Holt, P. F., and King, D. T., *Nature*, 175, 514 (1955).

Radioactivation Analysis of Phosphorus in Silicon

In a previous communication¹, a radioactivation method for the determination of arsenic in 'high purity' silicon was outlined. Further work on trace impurities in this element has resulted in the development of a technique for estimating submicrogram quantities of phosphorus.

On irradiating a silicon sample with thermal neutrons, the following reactions take place.



It can be seen that phosphorus atoms are produced by the decay of silicon-31 and that these are then activated according to equation (2). As these phosphorus-31 atoms are produced exponentially during irradiation, they are subjected to varying irradiation times and so the activity they produce, when extrapolated to zero time, merely indicates an apparent phosphorus concentration. The following expression has been derived for this concentration:

$$C_{\text{app}} = \frac{6\phi\sigma T \times 10^5}{0.69 N} \left\{ \frac{0.69 T_0}{(1 - \exp - 0.69 T_0/T)T} - 1 \right\}$$

in parts per million (3)

where ϕ is neutron flux, σ is cross-section of silicon-30 (barns), T is half-life of phosphorus-32 (sec.), T_0 is irradiation time (sec.), and N is Avogadro's number.

The above relationship indicates that the apparent phosphorus concentration varies as the neutron flux and irradiation time, so that irradiation conditions for phosphorus in the submicrogram range must be carefully chosen to give optimum analytical conditions. We have selected an irradiation time of one week at a pile factor of ten for routine determinations, when equation (3) gives 0.0011 p.p.m. of apparent

phosphorus, although under these conditions the total phosphorus-31 impurity introduced into silicon by radioactivation is 0.0024 p.p.m. (4.7×10^{13} atoms/gm. silicon).

We have used the following method for separating the active phosphorus chemically. Sufficient time is allowed to elapse after irradiation for the silicon-31 activity to have decayed to a negligible amount (one to two days for a sample of 0.5–1 gm.). The silicon is slowly dissolved in a hydrofluoric–nitric acid mixture with disodium hydrogen phosphate present as carrier, the rate of reaction being controlled by addition of nitric acid. After complete removal of the silicon as the gaseous silicon tetrafluoride, the solution is reduced to small bulk, perchloric acid is added and taken to fuming to remove hydrogen fluoride. Dilute hydrochloric acid is added to the solution and the phosphate present is precipitated as $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ with magnesia mixture and ammonia in the usual way². After standing for 24 hr. the precipitate is filtered, washed and redissolved with dilute hydrochloric acid. The magnesium ammonium phosphate is again brought down as before but in the presence of citric acid, and the whole is allowed to stand for a further 24 hr. It is then centrifuged, washed and slurried into a crucible and ignited at 1,100° C. to constant weight. The $\text{Mg}_2\text{P}_2\text{O}_7$ thus formed is finely ground and transferred to a counting tray to be counted and weighed.

Red phosphorus and disodium hydrogen phosphate were tried as standards. Known amounts were sealed in silica ampoules and irradiated with the silicon samples. They were dissolved and the phosphate extracted as already described. Although agreement was obtained between the two standards, disodium hydrogen phosphate was selected as the more suitable due to its higher purity and to the ease with which it could be dissolved.

Samples of 'pure' silicon from various sources, for use in semi-conductor devices, have been examined by this technique and found to contain phosphorus impurity ranging from 2.0 to 0.004 p.p.m. The distribution of phosphorus within a 'doped' single crystal has also been investigated by this method, and the results obtained are consistent with published segregation coefficients³, conductivity and autoradiographic measurements.

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¹ James, J. A., and Richards, D. H., *Nature*, 175, 769 (1955).

² Lundell, Hoffman and Bright, "Chemical Analysis of Iron and Steel", 218 (Wiley, 1931).

³ Burton, J. A., *Physica*, 20, 845 (1954).

Alpha-Oxidation of Omega-(2 : 4-Dichlorophenoxy)alkanenitriles and 3-Indolylacetone within Plant Tissues

BIOLOGICAL and chemical investigations carried out here on the plant growth-regulating activity of ω -aryloxyalkanecarboxylic acids have established the importance of β -oxidation as a mechanism by which these acids are degraded in plants¹. As a logical extension of this work, the corresponding nitriles were