Nature of the 'Induction Period' in the Preparation of Lithium Aluminium Hydride

For the preparation of lithium aluminium hydride, Schlesinger *et al.*[1] claim that an addition of lithium aluminium hydride to the ether covering the powdered lithium hydride is essential. In absence of it a smooth reaction between lithium hydride and aluminium chloride was not always possible, and a period of induction ranging from a few minutes to several hours followed. Such induction periods were undesirable as they made the reaction uncertain and also difficult to control once it began again. To overcome this difficulty, Schlesinger *et al.* give two elaborate methods for making lithium aluminium hydride for subsequent catalytic use. These methods unfortunately involve complicated apparatus which is difficult to find, if at all, in most laboratories.

Thus when making lithium aluminium hydride, owing to the non-availability of lithium aluminium hydride for catalytic purposes, the reaction was tried without the addition of lithium aluminium hydride to ether covering powdered lithium hydride. It was noticed that the reaction always started immediately and continued smoothly to the end. In view of these observations it was difficult to understand the difficulty encountered by other authors until the following clue was obtained.

Once, while making lithium aluminium hydride by the usual method (that is, without the addition of lithium aluminium hydride), the reaction failed to start and an induction period lasting for twelve hours followed. When the reaction was conducted on a portion of lithium hydride from the same source, but with an addition of lithium aluminium hydride to ether covering it, the reaction went smoothly. This proved conclusively that there was something in ether that was responsible for the induction period and that it was removed or destroyed by lithium aluminium hydride. A portion of the ether was therefore distilled over lithium aluminium hydride and then used in the preparation of hydride. The reaction went smoothly. The same effect was produced when ether was carefully dried again over sodium. In view of these results, it was clear that the induction period was caused by inactivation of lithium hydride powder, probably by water that remains in ether when not thoroughly dry. This view fits in well with the fact that, whereas sodium wire takes a long time to remove the last traces of moisture owing to its surface reaction alone, lithium aluminium hydride, by virtue of its solubility in ether, can remove moisture spontaneously on mixing. The inactivation of lithium hydride may therefore lie in the formation of a protective film, probably of lithium hydroxide, on the surface of its particles, and the induction period may well be the time required to remove this film by chemical and mechanical means. Once this unreactive coating is removed, a vigorous reaction is conceivable in presence of a large amount of aluminium chloride in ether.

In view of these observations, it can be stated that it is desirable to add lithium aluminium hydride for catalytic purposes; but in absence of it, a smooth reaction can be ensured by using carefully dried ether.

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[1] Schlesinger *et al.*, *J. Amer. Chem. Soc.*, **69**, 1199 (1947).

Intensification of the Latent Photographic Image produced by Beta-Rays

It is known that a latent image produced by a brief exposure to light can be intensified by a post-exposure to light of low intensity. This fact can be explained by assuming the production of a non-developable latent sub-image in addition to the normal latent image which can be developed. The latent sub-image can be converted to a developable latent image by a carefully controlled post-exposure to light of low intensity.

As the photographic action of charged particles on an emulsion is basically the same as that of light, it is to be expected that a latent sub-image is also produced by these particles. It is also to be expected that this latent sub-image would be produced in quantity when a fine-grain emulsion is exposed to fast particles of low charge, for example, if β-rays fall on plates which are not very sensitive to these rays.

We had available Kodak *NT* 2 *A* plates and a source of 0·01 μC. radium-D from the Atomic Energy Research Establishment, Harwell. This source was filtered by 10 mgm./cm.^{2} aluminium, so that only the β-rays from radium E were effective. The middle part of the plates was exposed to the β-rays for 1–2 days. After this exposure, half the plate was exposed to light from a 6·5-V. 0·2-amp. lamp at a distance of 2 m. for 20–28 sec. The plates were developed with *D* 19.

The density produced by the post-exposure alone varied from 0·007 to 0·11. For all post-exposures an increase of density of 50–100 per cent over that without post-exposure was obtained. Pre-exposure gave no appreciable effect. This agrees with the general rule that intensification can only be obtained if a long low-intensity exposure follows a short high-intensity exposure. The exposure of a grain hit by a β-ray is, of course, very intense and short in time.

Fuller details of this work will be published in due course in the *Revue de la Faculté des Sciences de l'Université d'Istanbul.*

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Decay of Light to Very Low Levels from Spark Discharges

Recent publications on the after-effects from the electrical explosion of wires[1] and in Geiger counters[2], the cold emission of electrons in spark gaps[3] and the light output of sparks[4] suggest that the following experiments may be of interest, as we have not seen comparable results reported elsewhere.

We have observed the decay of light from pulsed high-current discharges in several gases and liquids down to extremely low levels and extending for several hundred microseconds. Single, approximately rectangular, current pulses obtained from a condenser network charged to 10 kV., of 1–9 μsec. duration and 100–10,000 amp. peak value, were used with electrode separation of a few millimetres. Triggering was effected by means of a trigger pulse applied to an auxiliary electrode. The light output was detected by means of an *RCA* 931*A* photomultiplier cell, amplifier and oscillograph. By altering the high