



Fig. 1

intramolecular platinum-oxygen distances range upwards from 2.89 Å, more than 1 Å greater than is found in the example of the oxygen-chelated acetylacetonate anion in  $K Pt(Acac)_2Cl$ <sup>1</sup>. The mean lengths of the bridging and remaining C—O bonds are 1.44 and 1.40 Å respectively (each with  $\sigma$  0.03 Å).

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<sup>1</sup> Figgis, B. N., Lewis, J., Long, R. F., Mason, R., Nyholm, R. S., Pauling, P. J., and Robertson, G. B., *Nature*, **195**, 1278 (1962).

<sup>2</sup> Oldham, C., and Lewis, J. (in the press).

<sup>3</sup> Allen, G., Long, R., Lewis, J., and Oldham, C., *Nature*, **202**, 589 (1964).

### Ultra-purification by Separation of Aerosol Particles

PURIFICATION methods which treat the concentration of a given impurity as a continuous quantity become ineffective when only a discontinuous distribution of that impurity is left in a material. Further purification is feasible on a dispersion principle which makes use of this discontinuity, and by which liquids have been observed free from particles at temperatures too low to cause the latter to be melted or dissolved.

A bulk sample of liquid, which after thorough pre-purification still contains a number of particles, is partitioned into a larger number of droplets which are usually suspended in a particle-free fluid. In the resulting dispersion, the particles are confined to a fraction of the droplets and thus isolated from the rest of them<sup>1,2</sup>.

Liquid dispersions of metals<sup>1-4</sup>, water<sup>5-7</sup>, organic compounds<sup>8,9</sup>, and alkali halides<sup>10</sup>, of uniform droplet size, were subjected to under-cooling. Meanwhile, the droplets were observed either collectively or singly; their solidification was detected by inoculation of under-cooled bulk liquid<sup>5</sup>, by optical effects, for example, recalescence flashes<sup>8</sup>, twinkling<sup>8,10</sup>, or X-ray diffraction<sup>4</sup>, and by other techniques. A portion of the droplets did not freeze until, far below the melting point, the critical under-cooling temperature had been reached. There is evidence that these droplets crystallized by homogeneous nucleation and therefore were free from heterogeneous impurities. Homogeneous impurities did not alter very much the critical under-cooling temperature<sup>8</sup>, unless, as in emulsions, they were occluded in the particles<sup>9</sup>.

The isolation of particle-free liquids was always incidental to the investigation of other topics and the possibility of using it to produce these liquids in appreciable quantity does not seem to have been realized. For example, a drop (2 mm diameter) of particle-free water, stored at the interface between two immiscible liquids, was instead obtained by multiple distillation without ebullition<sup>7</sup>.

Large samples of liquids free from particles could be produced from aerosols by separating contaminated from uncontaminated droplets. When the temperature of a monodisperse liquid aerosol is held slightly above the critical under-cooling point, all droplets containing particles are frozen. By ultra-sound of a wave-length comparable with the diameter of these droplets, the particle-free liquid droplets can then be sub-divided further. The resulting difference in particle size causes the contaminated particles to sediment before the uncontaminated ones.

Bulk samples of materials could similarly be purified from the last traces of a given homogeneous impurity. The properties of the latter determine the technique by which the aerosol particles are separated. In the case of impurities amenable to uninterrupted selective heating, it is advisable to utilize the very large negative temperature coefficient of the rate of crystallization of particle-free droplets. The noble gas, in which the monodisperse liquid aerosol is suspended, is cooled to the critical under-cooling temperature. Then only the contaminated droplets, which are subjected to induction or to high-frequency dielectric heating, remain liquid and can be dispersed ultrasonically. In other cases, for example, contaminated aerosol particles could be photo-ionized selectively by ultra-violet radiation, and electrostatically precipitated.

According to the dispersion principle, the fraction of particles free from impurities increases with the degree of dispersion. Volatile covalent compounds can be dispersed to discrete molecules within an involatile inert matrix. This makes it possible to purify small quantities of such compounds by fractional molecular distillation, in which these molecules are separately evaporated by selective infra-red radiation<sup>11</sup>. The high cost of this method limits it to laboratory use.

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### Polyamide Layer Chromatography of Oestrogens

THE oestrogens were investigated by Struck<sup>1</sup> and Lisboa *et al.*<sup>2</sup> by thin-layer chromatography using silica-gel. The solvent systems for the silica-gel thin-layer were well-established ones which have been used for quantitative purposes<sup>3</sup>. On the other hand, Woltz and Chattaraj<sup>4</sup> combined thin-layer and gas-chromatography to identify the minor oestrogenic substances in female urine.

Since oestrogens contain a phenolic group in their molecule, it is conceivable that polyamide can be used