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Tritolyl phosphate—a suitable immersion oil for fluid inclusion freezing-stage studies

INFORMATION on the bulk composition and salinity of fluid inclusions in minerals can be obtained from the direct observation of various phase changes in frozen or cooled inclusions using a microscope freezing-stage (for example, see Roedder, 1963; Hollister and Burruss, 1976). Transient phases that develop on cooling such as ice, solid CO_2, CO_2-hydrate, and NaCl-hydrate are, however, often very difficult to observe because of the small size of most fluid inclusions (generally less than 100 μm). To facilitate these observations and to enhance the definition of small inclusions in transparent minerals it is normal practice to prepare high-quality, polished wafers of the host mineral. A relatively simple preparation technique has recently been published by Brumby and Shepherd (1978), but situations commonly arise where observations at low temperatures are required on inclusions in small mineral grains or fragments unamenable to routine polishing procedures. This note describes an alternative method of improving the optical quality of such material by immersing the sample in a small drop of tritolyl phosphate (tri-cresyl phosphate or phosphoric acid tri-(methyl-phenyl) ester).

Tritolyl phosphate, a readily available industrial chemical marketed under the trade names Lindol, Celluflex, and Kronitex, exists in three isomeric forms (ortho-, meta-, and para-cresyl; M. wt. = 368.36). The ortho form, tri-*o*-cresyl phosphate, has certain toxicological hazards particularly following ingestion (for details see Sax, 1975, p. 1190) and is usually excluded as much as possible from commercial tritolyl phosphate. However, it is advisable to specify 'ortho-free' when ordering this substance.

Ortho-free tritolyl phosphate is a suitable immersion oil for quartz at room temperature (Gordon, 1945). It is a colourless, odourless, and viscous oil, stable at ambient temperatures, has a refractive index of 1.55, is cheap to purchase, and, provided the usual laboratory precautions are taken, is safe to handle.[1] But the particular quality that makes it ideal for low temperature fluid inclusion studies is that *it maintains its* transparency at sub-zero temperatures (down to at least −100 °C). Other readily available oils with refractive indices in the range 1.5 to 1.6 such as clove oil, bromoform, and chlorobenzene have been tested in our laboratories but either freeze to a multigranular, crystalline aggregate or become progressively translucent on cooling. Tritolyl phosphate does not crystallize but freezes to a transparent metastable glass (the pour point of commercial-grade tritolyl phosphate is −28 °C according to the 1976 Merck Index).

[1] Soap and water are sufficient to remove this substance from surfaces. However, tritolyl phosphate is a well-known plasticizer and any contact with vinyl plastics should be avoided.

TABLE I. *Comparison of the results obtained for the last ice-melting temperature* (± 0.3 *°C*)* *of three primary gas/liquid inclusions in quartz from a quartz-cassiterite vein, St. Michael's Mount, Cornwall, using the two methods described in text.*

Inclusion	Last ice-melting temperature (°C)	
	Immersion method	Polished wafers
A	−2.8	−2.8
B	−2.7	−2.8
C	−2.8	−2.9

* All measurements were carried out on a modified version of the freezing-stage described by Smith (1973).

Results obtained for the temperatures of phase changes in frozen inclusions using this immersion oil are identical, within the limits of experimental error, to those obtained for the same set of inclusions using conventional polished wafers (Table I). Crystallization of the glass does not occur even when the cell is maintained at temperatures below −60 °C for several hours.

Whilst tritolyl phosphate is ideal for low temperature inclusion studies in quartz grains because of their similar refractive indices it has also proved effective for similar studies in our laboratories on minerals possessing higher or lower refractive indices such as apatite, calcite, and fluorite.

Our usual procedure is to place a few drops of tritolyl phosphate together with a sprinkling of the test material (mineral grains, fragments, etc.) on a clean microscope slide under an ordinary petrographic microscope. Suitable grains are selected and transferred to a small, 0.3–0.5 cm^2, cover slip. Transfer can be effected either by using a finely drawn, glass tube or fine-pointed, steel probe. The cover slip is then placed in the microscope freezing cell after ensuring that sufficient oil covers the mineral fragment.

Fragments up to about 1 mm in size can be accommodated in this way; larger samples can be used but care must be exercised to minimize the effects of these larger thermal masses by careful calibration of the apparatus and very slow cooling and heating rates.

Unfortunately, tritolyl phosphate is unsuitable for routine use as an immersion oil for fluid inclusion heating-stage studies because of its gradual decomposition above about 200 °C to the highly toxic vapour $(PO)_x$ (Sax, 1975). Other oils with suitably high boiling points such as high molecular weight paraffins and silicones may, however, be used.

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