X-Temp 2

Low Temperature Single Crystal Handling for X-ray Structure Analysis A Combination of Crystal Cooling and Oil-Drop Mounting Technique



Controller

Evaporator

Insulation Container

contact address

Prof. Dr. D. Stalke Institut für Anorganische Chemie der Georg AugustUniversität Tammannstraße 4 37077 Göttingen

 Tel. (int.)
 +49-(0)551-39-3000 or 3045

 FAX (int.)
 +49-(0)551-39-3459

 email
 dstalke@chemie.uni-goettingen.de

 web
 www.stalke.chemie.uni-goettingen.de

In combination with inert oils (e.g. perfluorinated polyethers FOMBOLIN YR 1800 and GALDEN HT 90 from AUSIMONT DEUTSCHLAND GMBH, Kölner Straße 3a, Postfach 5202, D-65760 Eschborn (www.ausimont.de))

X-Temp 2 facilitates

- handling of highly reactive and/or thermolabile compounds (e.g. the solid state structure of n-butyllithium T. Kottke, D. Stalke *Angew. Chem. Int. Ed. Engl.* **1993**, *32*, 596.)
- monitoring a reaction pathway by means of low temperature X-ray structure analysis (A. Heine, D. Stalke Angew. Chem. Int. Ed. Engl. 1992, 31, 854.)
- crystal preparation (selection, cutting, washing, mounting at the tip of a glass fiber) at any constant temperature between room temperature and -120°C (D. Stalke *Chem. Soc. Rev.* 1998, 27, 171.)
- investigation of the crystal quality by the polarization device of any commercial microscope.
- 3-5 hours operation time with a single filling and pressureless top-up without the need of interrupting low temperature work.

Recent results achieved employing *X*-*Temp 2*

- Ulrike Flierler, Michael Burzler, Dirk Leusser, Julian Henn, Holger Ott, Holger Braunchweig, Dietmar Stalke "Electron Density Investigation of Metal–Metal Bonding in the Dinuclear "Borylene" Complex [{Cp(CO)₂Mn}₂(μ-B^tBu)]" Angew. Chem. Int. Ed. 2008, 47, 4321.
- 2. F. N. Shi, L. Cunha-Silva, R. A. Sà Ferreira, L. Mafra, T. Trindade, L. D. Carlos, F. A. Almeida Paz, J. Rocha "Interconvertable Modular Framework and Layered Lanthanide(III)-Etidronic Acid Coordination Polymers" *J. Am. Chem. Soc.* **2008**, *130*, 150.
- 3. Hanne Nuss, Martin Jansen $Cs_5([12]crown-4)_2(O_3)_5$: A Supramolecular Compound Containing the Confined Ozonide Partial Structure $\{Cs_8(O_3)_{10}\}^{2}$. *Angew. Chem. Int. Ed.* **2006**, *45*, 7969.
- 4. Hanne Nuss, Martin Jansen "[Rb([18]crown-6)(NH₃)₃]Au·NH₃: Gold as Acceptor in N_H···Au-Hydrogen Bonds" *Angew. Chem. Int. Ed.* **2006**, *45*, 4369.

Specification of X-Temp 2

a) model

evaporator: nickel plated brass; recooling of evaporated nitrogen to guarantee the gas stream temperature being independent from the liquid nitrogen level

top of insulation container: aluminum/polystyrene material

main heater: ceramic power resistors

transfer line: two silvered glass tubes with vacuum jacket, interconnection by a teflon adapter and a screw cap with silicon sealing; integrated heating coil (stream heater)

nozzle: teflon material with fan-shaped outlet, integrated nozzle heater, connected in series with the main heater, integrated thermosensor.

controller: illuminated switches, triac controlled power supply of heating elements, visual and acoustic alarm at low nitrogen level, automatic shut-off of all heating elements at minimum level, external miniature thermosensor, operating current 110/220 V switchable

b) technical data

liquid nitrogen capacity: 5 L

operation time: 3-5 h (dependent on gas flow rate) per single filling, refillable at standard pressure without interrupting operation

T(min): -120°C, T(max): 30°C, temperature constancy Δ T better than \pm 2°C

c) dimensions

controller: 110 x 290 x 200 (H x W x D in mm), weight 4 kg *evaporator:* 250 x 390 (\emptyset x H in mm), weight 9 kg *height of transfer line:* 200 mm above top of insulation container *length of transfer line:* 220 mm from edge of container to top of nozzle outlet

Quote for the X-Temp 2 system package

 liquid nitrogen evaporator with thermo insulation container
 transfer line with integrated stream heater, teflon adapter and screw cap
 teflon nozzle with built in nozzle heater and thermosensor
 controller for generating and regulating the nitrogen cold gas stream, operating at 110/220 V power supply
 external thermosensor

X-Temp 2 : System for Crystal Handling at Low Temperatures



crystal cooling by a N₂ inert gas stream generated by X-Temp 2 easy crystal handling under the microscope down to -120° C without icing



Single crystals immersed in an inert oil X-Temp 2 cooled on the microscope slide