Sodium Hydride 60% Dispersion in Oil

Description

Sodium hydride is used as a deprotonating agent in condensation and alkylation reactions, and as a polymerization catalyst.

Sodium hydride is insoluble in most solvents, including ethers, hydrocarbons, amines and ammonia. It is offered by Rohm and Haas as a 60 ± 3% NaH microcrystalline dispersion in mineral oil. The particle size range is typically 5-50µ.

Typical Physical Properties

These properties are typical but do not constitute specifications.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>NaH</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>24.00</td>
</tr>
<tr>
<td>Form</td>
<td>Microcrystalline dispersion of gray powder in mineral oil.</td>
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<tr>
<td>Specification</td>
<td>NaH: 60% ± 3%</td>
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<tr>
<td>Diluent</td>
<td>Mineral oil: 40% ± 3%</td>
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<td></td>
<td>The mineral oil is freely miscible with a wide range of hydrocarbons not affecting the activity of sodium hydride. Examples include hexane, heptane, diethyl ether, dibutyl ether and toluene.</td>
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<tr>
<td>Viscosity</td>
<td>75-85 sus @ 100°F</td>
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<tr>
<td>Flash point</td>
<td>340°F</td>
</tr>
<tr>
<td>Specific Gravity</td>
<td>0.84-0.86 (25°C)</td>
</tr>
</tbody>
</table>

Pure sodium hydride dissociates on heating, starting at about 225°C. The relationship of equilibrium pressure to temperature is expressed by the equation:

\[
\log_{10} P = \frac{-6100}{T} + 11.66^{(1)}
\]

where \( P \) = pressure in mm Hg \( T \) = temperature in K

(1) Herold, Compt. rend 228, 686 (1949)

As for the dispersion in mineral oil, NaH starts deprotonating oil at temperatures above 300°C.

Water Reactivity

CAUTION: Upon reaction of sodium hydride dispersion with water, \( H_2 \) and NaOH are formed. This hydrolysis reaction is vigorous and exothermic.

Application

Condensations

Sodium hydride in oil resembles sodium and sodium alcoholates in its ability to function as a deprotonating agent in the Acetoacetic ester, Claisen, Stobbe, Dieckmann, and related condensations. It possesses marked advantages over other condensing agents in that:

1. It is a stronger base, which results in a more straightforward deprotonation
2. No excess is needed
3. Alcoholates add an alcohol to the reaction mixture, which may need to be distilled out to drive the condensation equilibrium – NaH results in only \( H_2 \) gas as a reaction by-product
4. The evolving \( H_2 \) gives a measure of the extent of the reaction
5. Side reactions such as reductions are eliminated
Alkylations

Alkylations of aromatic and heterocyclic amines such as 2-aminopyridine and phenothiazine are readily accomplished in high yield using toluene-dimethylformamide mixtures. The dimethylformamide concentration is a variable used to control the rate of reaction.

Polymerization

Several literature examples indicate the usefulness of sodium hydride in polymerization to form caprolactams and glycol ethers.

Product Stewardship

Rohm and Haas’ 60% NaH dispersion is not pyrophoric, but it has some inherent handling risk due to its ‘Dangerous When Wet’ classification. Therefore, Rohm and Haas sells this product as part of a comprehensive Product & Services package, which includes:

- The highest NaH product quality
- A formulation in mineral oil which is stable under various transport conditions
- The availability of a choice of solvent soluble bags and package sizes
- Safety audits and training
- Technical advice with regards to both the safe handling and the cost-efficient synthetic use

Availability/Packaging/Shipping

Sodium hydride dispersion is available in 15 kg and 90 kg drums containing individual 5 kg units, which are packaged in the solvent soluble bag of choice. Two 5 kg units are then packed together in a polyethylene bag, which is placed in friction top cans or metal pails.

Special packaging is available to meet specific process needs.


Toxicity and First Aid

Sodium hydride dispersion is considered to be toxic if ingested due to the caustic formed by reactions with water, and to possible gas embolism resulting from reaction with stomach acids. In case of ingestion, do not induce vomiting. Contact a physician immediately and transfer the person to a medical facility.

Sodium hydride dispersion is considered corrosive to skin or eyes, also because of the potential for caustic byproducts from reactions with moisture. In case of eye contact, flush with large amounts of water, under eyelids for at least 15 minutes, and seek medical attention immediately thereafter. In case of skin contact, brush off immediately and flush the affected area with water. Seek medical attention if irritation persists.

Sodium hydride dispersion is non-dusting. However, reacting material can emit a fine caustic mist. In case of inhalation, rinse mouth with water and move to fresh air. Seek medical attention immediately.

All precautions should be taken against ingestion, inhalation and eye or skin contact with sodium hydride dispersion or reaction products. Refer to the Material Safety Data Sheet for additional information or contact Rohm and Haas Company.

Handling and Storage

Sodium hydride dispersed in oil is not a pyrophoric material, but material adhering to covers and side walls of containers may ignite when exposed to humid air. Therefore, protective rubber gloves, clothing and face shields should be worn when handling sodium hydride. The dispersion should be handled in the same manner as other hygroscopic materials. Store in a clean dry area and keep containers tightly closed. Transfer of hydride to other containers should be done only after container has been baked dry, cooled and flushed with nitrogen. Sodium hydride reactions should always be carried out in adequately vented vessels.

Rohm and Haas’ dispersion of 60 ± 3% NaH is a safe stable material to handle. Still, depending on the transport and storage conditions, some mineral oil may drain away from the hydride particle on standing resulting in a dispersion on top of the container which must be handled with caution as indicated.
**Fire Precautions**

Sodium hydride dispersion is classified ‘Dangerous When Wet’. Upon reaction with water, hydrogen gas and sodium hydroxide (\(H_2 + NaOH\)) are formed. This hydrolysis reaction is vigorous and exothermic. When not vented appropriately, there is a risk of \(H_2\) ignition.

*CAUTION: POWDERED LIMESTONE FOR SMOTHERING FIRES SHOULD BE AVAILABLE. DO NOT USE WATER, SODA ACID, CHEMICAL OR CARBON DIOXIDE EXTINGUisher. THE ONLY RECOMMENDED FIRE EXTINGUisher IS THE NITROGEN PROPELLED DRY POWDER TYPE. REFER TO THE MATERIAL SAFETY DATA SHEET FOR MORE DETAILS.*