2nd EUROPEAN CONFERENCE ON PLANT & PROCESS SAFETY
September 13 & 14, 2022
Flanders Convention Centre, Antwerp, Belgium

TRIPHOSGENE
SAFE HANDLING IN INDUSTRIAL ORGANIC PROCESSES

Livius Cotarca
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Livius Cotarca

PhD, Industrial Organic Chem and Chem Eng

- Seasoned professional with over four decades of experience in the Organic Process Research and Development, Pharmaceutical, Biotech, and Life sciences, Global Operations, and Supply Chain.
- Particular area of interest is the synthesis of Active Pharma Ingredients (APIs), Fine Chemicals and Performance Organic Intermediates and Products by using either new synthetic routes or innovative reagents, e.g., **Triphosgene**.
- Considerable track record of leading positive, collaborative teams that get things done.

Current Position: Owner and General Manager at LC CONSULTING SAS, Udine, Italy, providing expertise in

- Process research and development for Active Ingredients (API) and advanced intermediates, and
- Innovative business models in pharmaceutical solid-state characterization *by Synchrotron radiation*

Work Experience:

- Senior Scientist and Group Leader at **SNIA – CAFFARO Group**, Italy
- Global R&D Director of Italian Pharma Company, **Zambon Group spa**
- Co-Founder and Director of **ISPROCHEM** (International School of Process Chemistry)
- Associate Professor of Organic Chemistry and Co-Founder of the **Master** in "*Process Chemistry in Pharma Industry*" – University of Milan "*La Statale*", Italy

Co-Authorship:

- 80 + developed processes for APIs and advanced intermediates; 60 + families of patents on pharma processes for active ingredients; 3 books (*Phosgenation Handbook/Structure – Properties Relationship in Organic Chemistry*); 60 + papers in the field of organic and physical organic chemistry of technological importance;

*“SO GIVE TO CAESAR WHAT IS CAESAR’S….”**, PHOSGENE: ESSENTIAL IN EVERYDAY PRODUCTS

PHOSGENE IS A RAW MATERIAL USED PRIMARILY IN THE PRODUCTION OF TWO OTHER CHEMICALS:

METHYLENEDIPHENYLDIISOCYANATE (MDI), AND

TOLUENE DIISOCYANATE (TDI).

PHOSGENE IS PRODUCED BY COMBINING CARBON MONOXIDE AND CHLORINE WITH A CATALYST.

PHOSGENE IS ESSENTIAL IN THE MANUFACTURING OF PRODUCTS USED IN EVERYDAY LIFE:

FLEXIBLE FOAMS IN UPHOLSTERED FURNITURE,

RIGID FOAMS AS INSULATION IN WALLS AND ROOFS,

THERMOPLASTIC POLYURETHANES USED IN MEDICAL DEVICES AND FOOTWEAR.

PHOSGENE IS IMPORTANT IN MANUFACTURING OF:

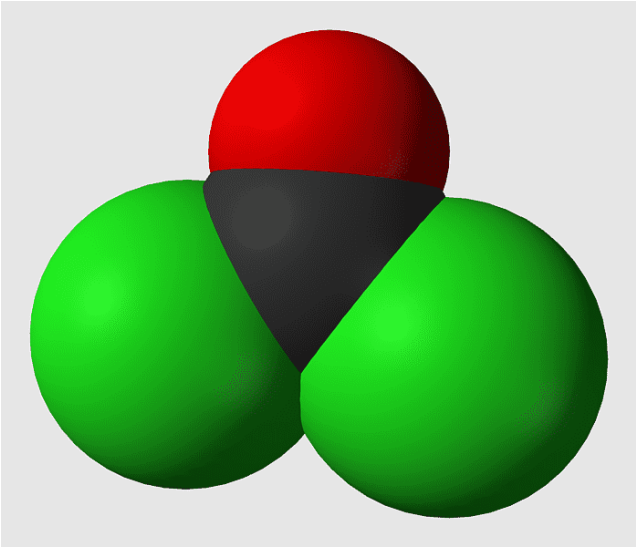
COATINGS, ADHESIVES, SEALANTS AND ELASTOMERS

POLYCARBONATE PLASTICS,

PHARMACEUTICALS,

AGRICULTURAL CHEMICALS, AND

SPECIALTY CHEMICAL INTERMEDIATES.



**“SO GIVE TO CAESAR WHAT IS CAESAR’S, AND TO GOD WHAT IS GOD’S.” – MATTHEW 22:21*

https://www.americanchemistry.com/industry-groups/phosgene

OUTLINE

Triphosgene (BTC) is a Chlorinated Organic Carbonate

Chemical reactivity and safety: “decomposition” to phosgene

Discovery literature and current publication flow

Sources/Manufacturing capacities/Supply quality

Relevant physical properties

Misleading literature

Toxicology

Stability and Monitoring

Users and Logistics: Packaging, Storage, Transportation, and Handling

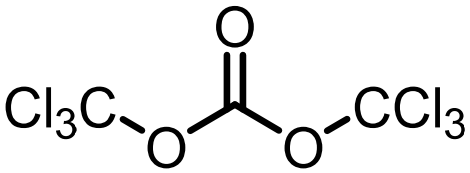
Safety of Industrial operations

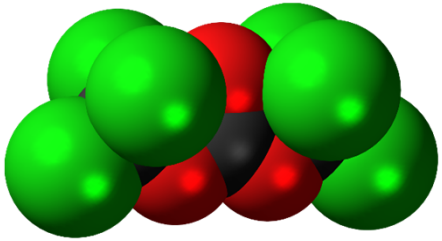
Conclusions and Outlook

TRIPHOSGENE – A MISNOMER

SYNONYMS: **B**IS(**T**RICHLOROMETHYL)**C**ARBONATE (**BTC**), “SOLID PHOSGENE”

$(COCl_2)_3$





MW 98,92 x 3 g/mol

MW 296,748 g/mol

NOT A LINEAR OR BRANCHED
TRIMER OF PHOSGENE

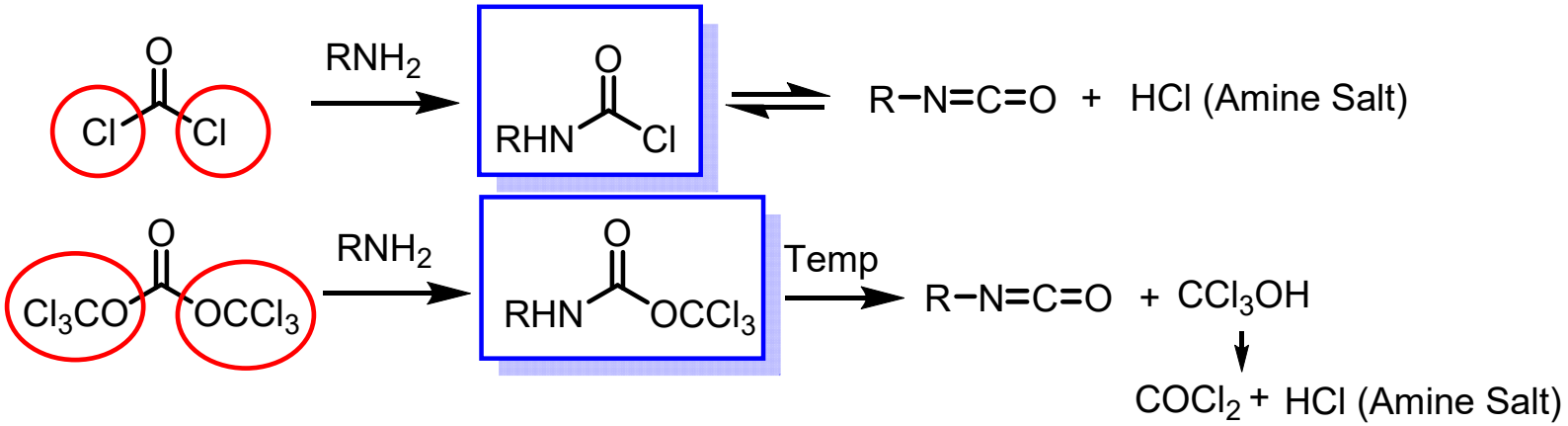
BTC IS MAINLY EMPLOYED IN THE FINE CHEMICAL INDUSTRY.

COMMERCIAL MANUFACTURE AND APPLICATIONS ARE PROMOTED DUE TO:

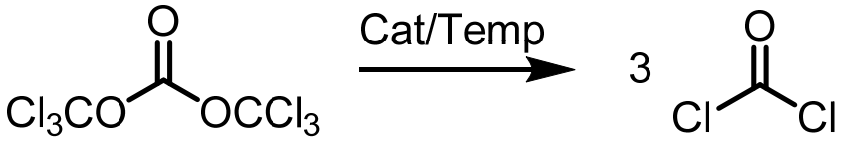
- LITERATURE (advertised as safe or safer phosgene)
- SOLID STATE (handling aspects)
- LEGISLATION (especially in China)

CHEMICAL REACTIVITY AND SAFETY

VERY SIMILAR BUT NOT IDENTICAL TO THAT OF PHOSGENE

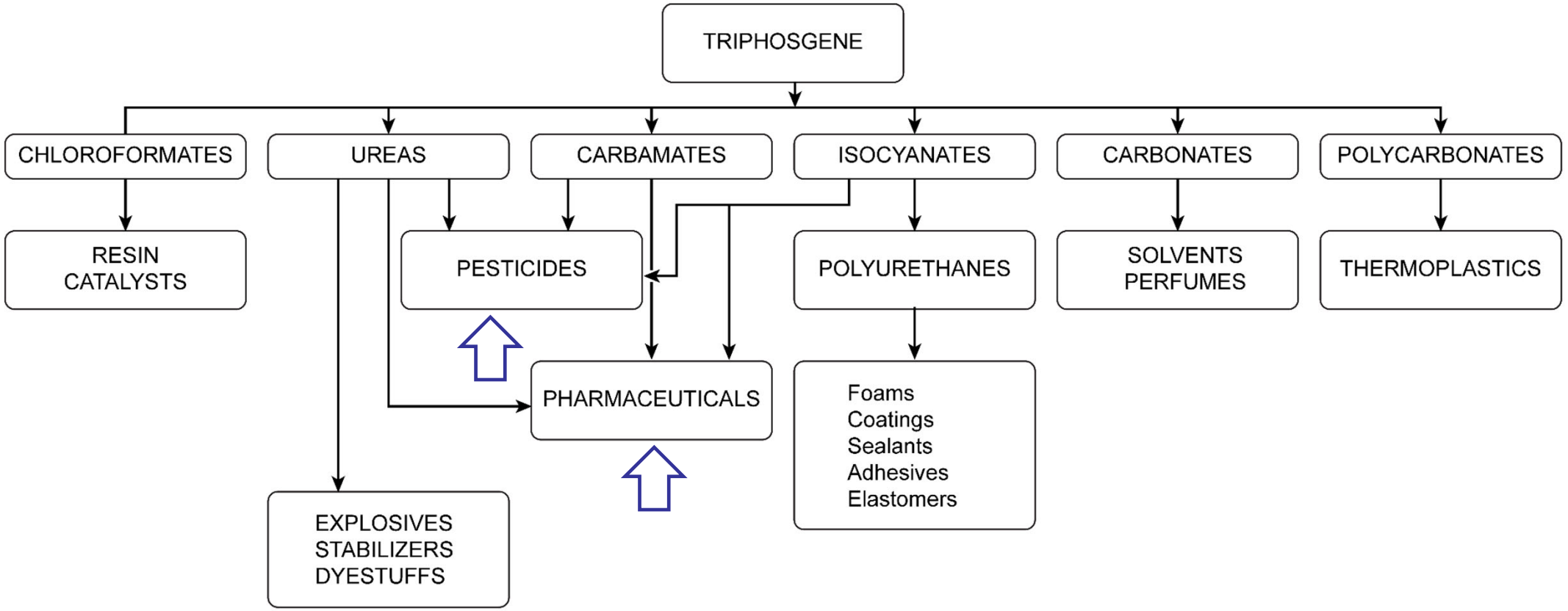


QUANTITATIVE "DECOMPOSITION" OF BTC TO PHOSGENE



THE USE OF TRIPHOSGENE RESULTS IN THE CO-EXISTENCE OF PHOSGENE AND TRIPHOSGENE IN THE LIQUID AND GAS PHASES OF INDUSTRIAL PROCESSES

PHOSGENATIONS BY TRIPHOSGENE



INDUSTRIAL USE OF TRIPHOSGENE IS RELEVANT IN THE FINE CHEMICALS INDUSTRY (PHARMACEUTICALS, AGRICULTURAL CHEMICALS, AND SPECIALTY CHEMICAL INTERMEDIATES).

ORGANIC PROCESS RESEARCH & DEVELOPMENT
OPR&D
Communication
Cite This: Org. Process Res. Dev. 2017, 21, 1439-1446
pubs.acs.org/OPRD

Bis(trichloromethyl)carbonate (BTC, Triphosgene): A Safer Alternative to Phosgene?

Livius Cotarca,[†,*] Thomas Geller,[‡] and József Répási[§]

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[‡]Product Supply, Crop Science Division, Bayer AG, 41539 Dorm
[§]SONEAS Hungary, Illatos út 33, Budapest, H-1097, Hungary

ABSTRACT: Bis(trichloromethyl)carbonate (BTC, triphosgene) is a versatile compound that enables highly efficient syntheses. In addition, because of its solid state, it is a very convenient compound for small-scale phosgenations. Consequently, this compound is favored as a phosgene substitute in research and development and in small-scale production. Although BTC is highly toxic, safe handling is possible as long as the properties and chemical reactivity of this compound are understood and considered. However, branding as "safe phosgene" or "safer phosgene" is misleading. The solid state of BTC leads to the misconception that there is no significant exposure. However, the vapor pressure is sufficiently high to easily result in toxic concentrations. In addition, proper monitoring is not yet possible. Proper use of BTC could be more complex than the handling of phosgene itself. However, handling of BTC is normally always associated with phosgene and has its own toxicity. Therefore, the use of BTC will become more regulated in the future, which will directly increase responsibility in route selection during process development. A stringent safety concept for phosgenations using BTC is necessary. Because of the interconnection with phosgene, the safety concept for BTC will likely be an extended version of the safety concept for phosgene.

Phosgene

ULLMANN'S ENCYCLOPEDIA OF INDUSTRIAL CHEMISTRY

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TRIPHOSGENE SAFETY REVIEW

Bis(trichloromethyl)carbonate (BTC, Triphosgene): A Safer Alternative to Phosgene?

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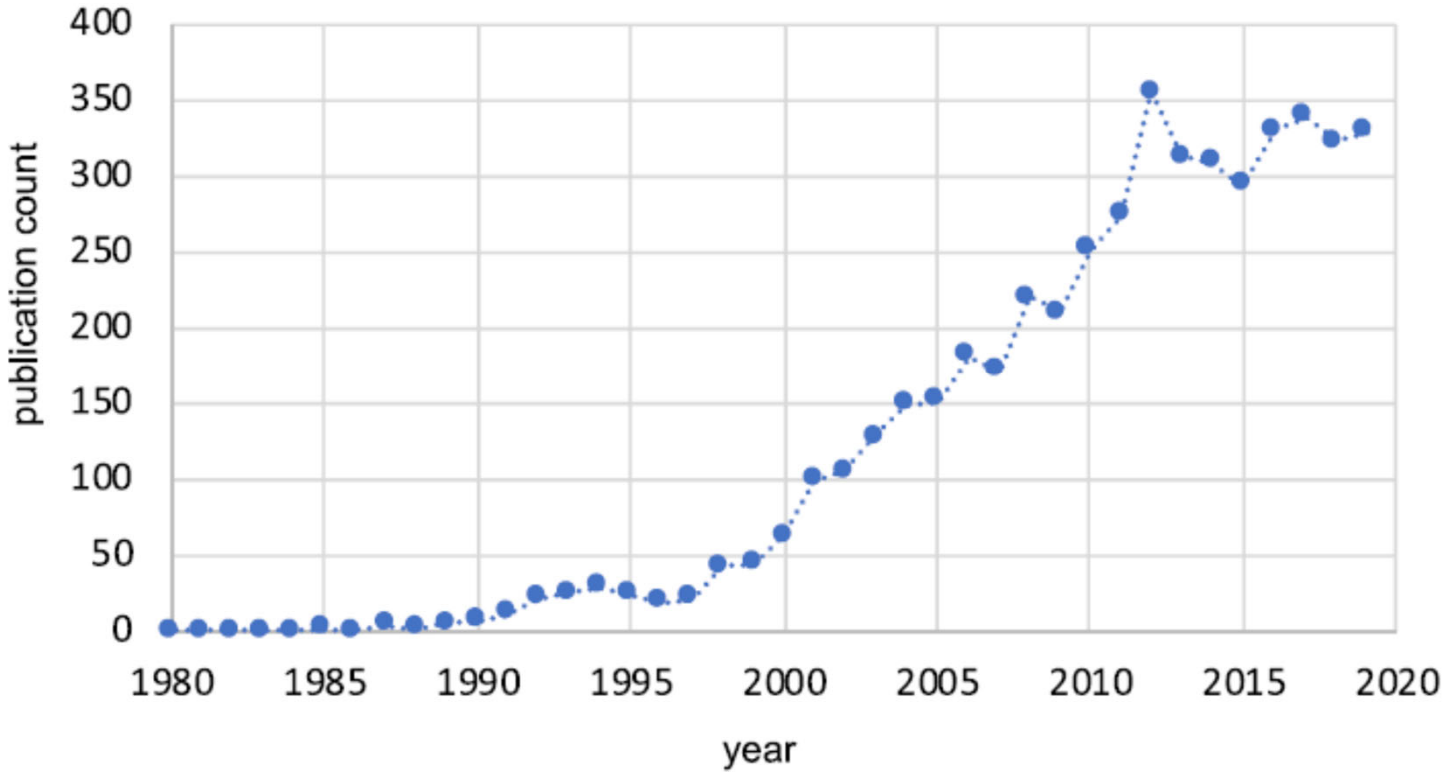
TRIPHOSGENE AND JOURNAL PUBLICATIONS TREND

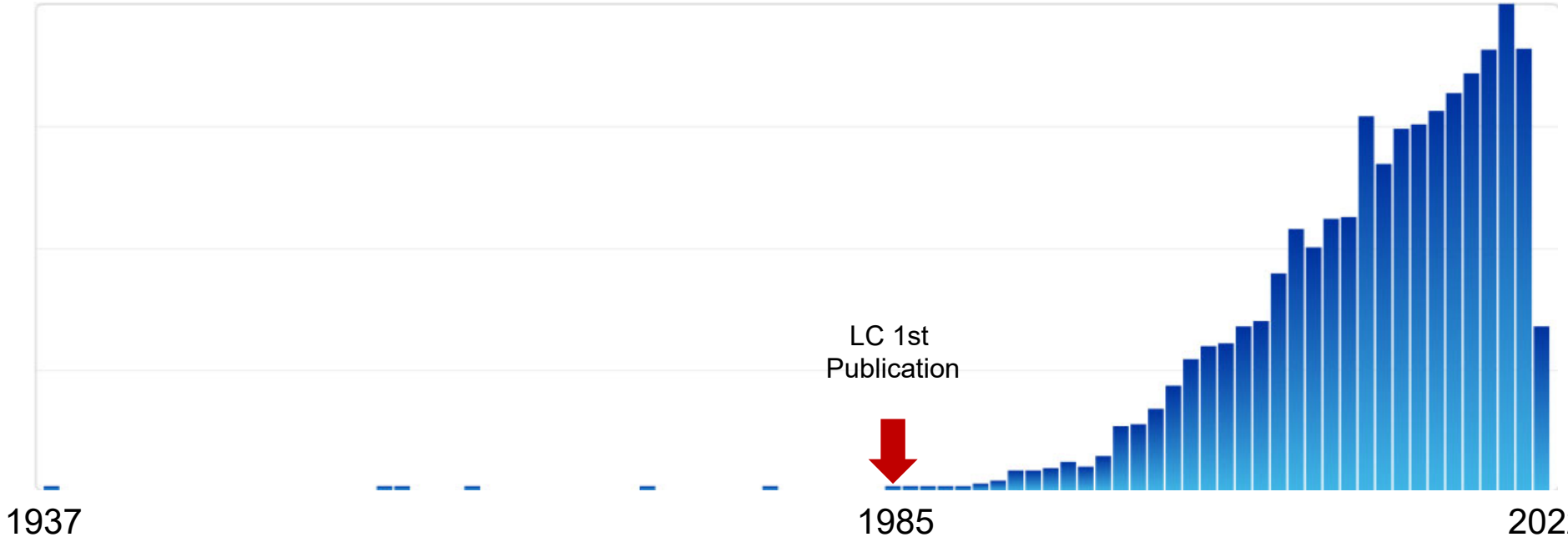


O. Ganiu, B. Nepal, J.P. Van Houten et al., A decade review of triphosgene and its applications in organic reactions, *Tetrahedron*, https://doi.org/10.1016/j.tet.**2020**.131553

DISCOVERY LITERATURE AND CURRENT PUBLICATION FLOW

7465 PATENTS/PATENT APPLICATIONS

5907 JOURNAL PUBLICTIONS
971 REF IN THE YEAR 2020



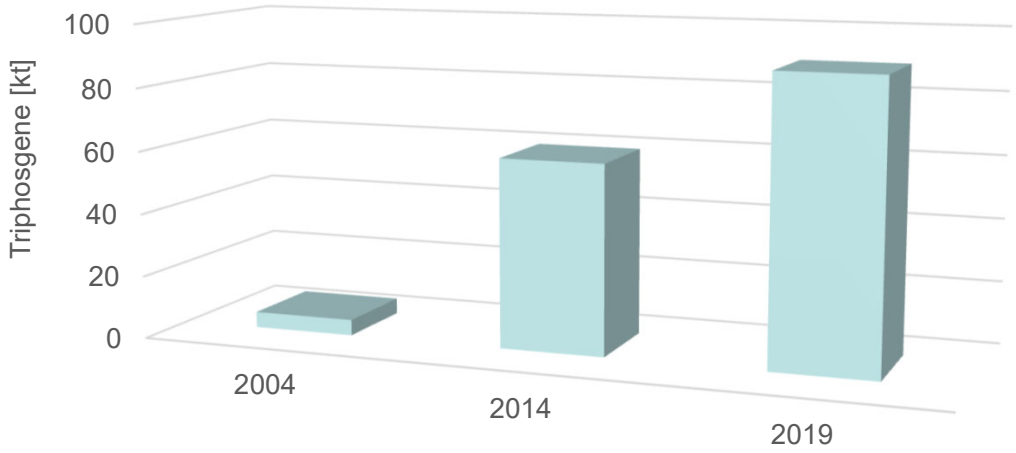
Source SciFinder® by July 5, 2022

FIRST DESCRIBED BY C. COUNCLER (1880)

LITERATURE INDICATES A GROWING INTEREST ON BTC

TILL NOW: 14.000 + REFERENCES IN THE LITERATURE!

TRIPHOSGENE MANUFACTURING CAPACITIES



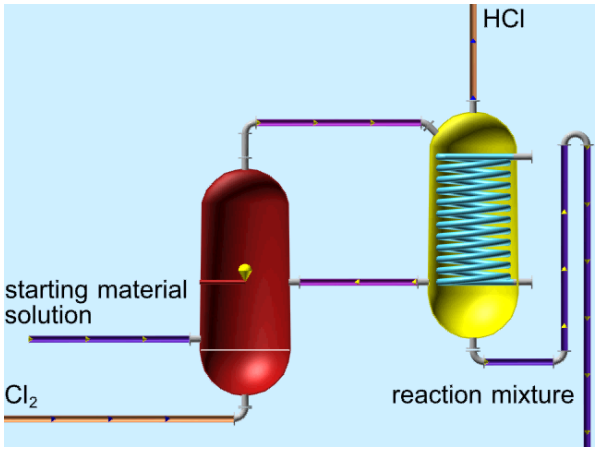
CHINA IS BY FAR THE LARGEST MANUFACTURER OF BTC

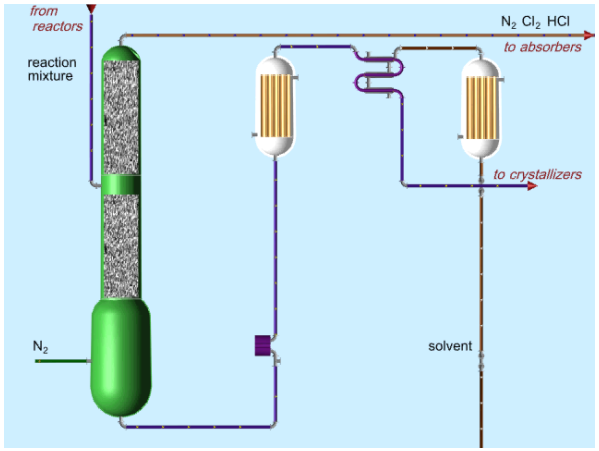
CAPACITIES 2022 : 100.000 + MT/Y (ESTIMATED)

MASSIVE CAPACITY INCREASE IN THE NEAR FUTURE IS EXPECTED (several large scale manufacturers & traders/distributors)

BTC IS MAINLY CONSUMED IN THE CHINESE DOMESTIC MARKET, ONLY A SMALL FRACTION IS EXPORTED (e.g. 2016: ca. 2000 T of BTC to India)

See references (1) and (2)

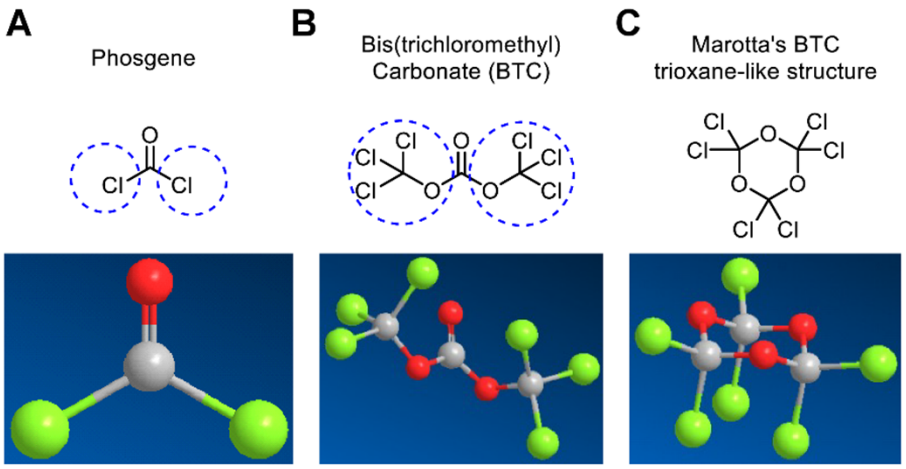


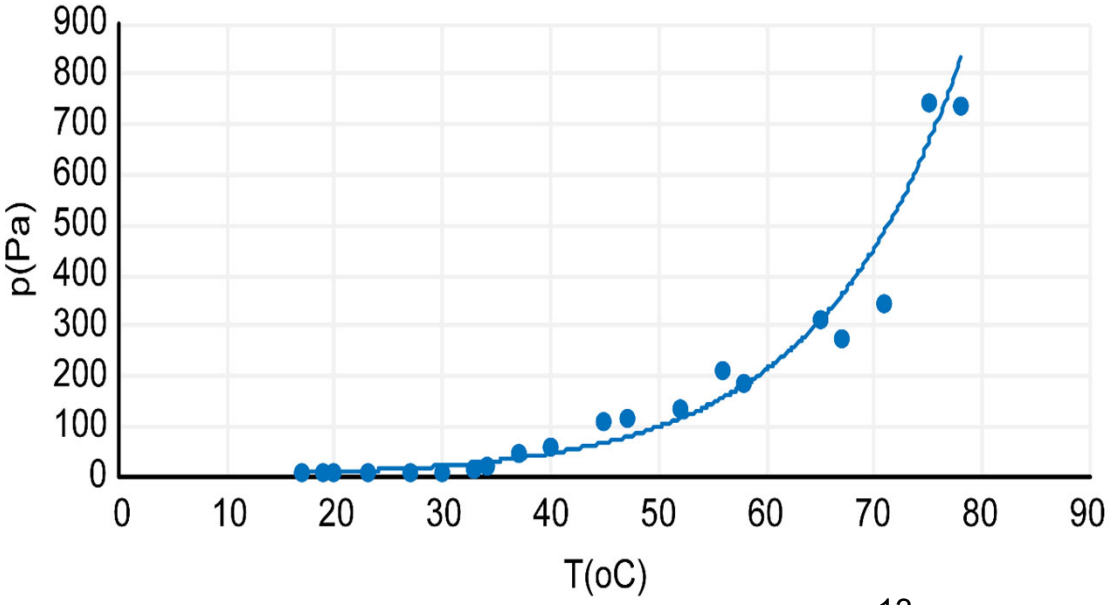


Triphosgene plant in HU
(shut down due to EU outshoring),
Ubichem, CD-ROM, 1999

BTC RELEVANT PHYSICAL PROPERTIES

Parameter	Phosgene	BTC
Phase @ 20 °C, 1 bar	colourless gas	crystalline colourless solid
bp [°C] (1013 mbar)	7.4 – 8.2	203 – 206
mp [°C]	-128 to -118	79 – 83
Density [g/cm^3]		1.723 (20 °C), 1.629 (80 °C)
Vapour pressure [mbar]	ca. 1590 (20 °C)	0.35 (25 °C)





Ubichem, Triphosgene, CD-ROM, 1999

TRIPHOSGENE PROPERTIES

PURE BTC IS STABLE; thermal decomposition starts slowly after melting (> ca. 95 °C)

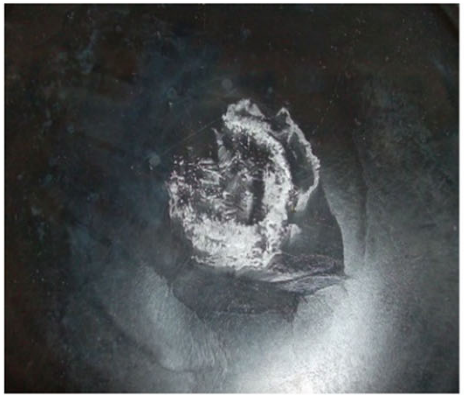
BTC QUALITY INFLUENCES THE STABILITY significantly (qualities on the market differ – even though high quality is always claimed!)

PURITY OF BTC IS NOT EASY TO DETERMINE; the use of the standard assay (chloride titration) is problematic

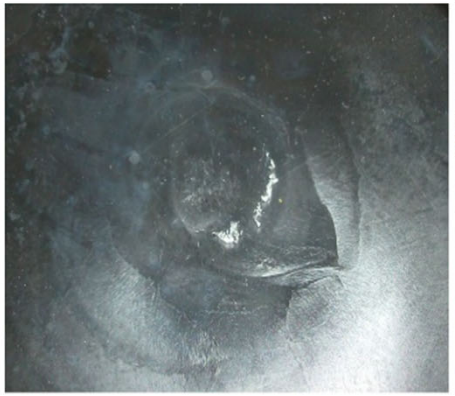
THERMAL DECOMPOSITION RESULTS IN FORMATION OF GASES (significant pressure buildup in closed systems; phosgene, CO_2, CCl_4; some decomposition pathways can also lead to formation of diphosgene!)

TRIPHOSGENE PROPERTIES

0 min



35 min



200 min

re-sublimation

BTC HAS A VAPOR PRESSURE COMPARABLE WITH **IODINE** AND ALSO A SIMILAR SUBLIMATION BEHAVIOR !

BTC VAPOR STREAMS CAN PASS THROUGH WATER WITHOUT REACTING (AND UNDER CERTAIN CIRCUMSTANCES ALSO THROUGH CAUSTIC!)

PURE SOLID BTC IS VERY HYDROPHOBIC (REACTION IS KINETICALLY HAMPERED)!

BTC DISSOLVED IN WATER MISCIBLE SOLVENT REACTS DIRECTLY WITH WATER!

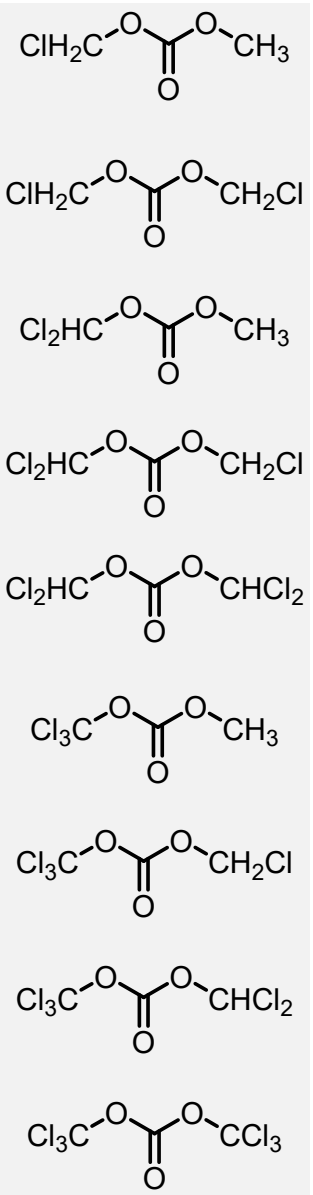
TRIPHOSGENE QUALITY

BTC OF INDUSTRIAL QUALITY CAN CONTAIN MORE THAN 20 DIFFERENT IMPURITIES AT CONCENTRATIONS GREATER THAN 0.1%.

PARTIALLY CHLORINATED BY-PRODUCTS (INTERMEDIATES OF THE BTC MANUFACTURE), METAL IONS, ACTIVATED CARBON AND NUCLEOPHILES (INCLUDING CHLORIDE ION!) CAN CATALYSE THE DECOMPOSITION!

IN THE MANUFACTURE OF APIs OR ADVANCED INTERMEDIATES, WHEN THE IMPURITY PROFILE OF THE PRODUCT MUST BE CONTROLLED, ^{1}H-NMR MUST BE USED.

ALTHOUGH IMPURITIES MIGHT BE ACCEPTABLE FOR THE PRODUCTION OF FINE CHEMICALS, THEY ARE NOT ACCEPTABLE FOR ACTIVE PHARMA INGREDIENTS (APIs) OR THEIR DIRECT PRECURSORS



MISLEADING LITERATURE

Triphosgene

From Wikipedia, the free encyclopedia

Triphosgene (**bis(trichloromethyl) carbonate** (**BTC**) is a chemical co… the formula $OC(OCCl_3)_2$. It is used as a safer substitute for phosgene, because, at room temperature, it is a solid, whereas … [5] Triphosgene decomposes above 200 °C.[6]

Contents [hide]

1 Preparation

Wrong!

Safety [edit]

The toxicity of triphosgene and phosgene are the … er decomposes to phosgene on heating and upon reaction with nucleophiles. Trace moisture leads to formation of p… herefore, this reagent can be safely handled if one takes all the precautions as for phosgene.[10]

07/11//2021: https://en.wikipedia.org/wiki/Triphosgene

MISLEADING LITERATURE

The "defusing" of dangerous and risky chemicals is exemplified here by the avoidance of the highly toxic, gaseous basic chem… sgene ($COCl_2$) as a reagent. …

*Bis(trichloromethy) carbonate **1** ("tripho… nd, is a crystalline, stable solid (m.p.= 80°C, b.p. = 20… light decomposition to phosgene, which is easy to…*

*A further advantage of **1** is that… …ly weighed out in milligram amounts. Furthermore, owing to t… …latility of **1**, only the usual safety precautions are necessary.*

Misleading!

H. Eckert, B. Forster; *Angew. Chem. Int. Ed.*, (1987) **26** 894

MISLEADING LITERATURE

... Darüber hinaus ergeben sich aber beim Arbeiten mit Triphosgen weitere überraschende Vorteile. [illegible] Triphosgen z.B. ohne Zersetzung bei 203 bis [illegible] destilliert werden. Es tritt ausserdem keine [illegible] konzentrierter Schwefelsäure oder [illegible] Wegen seiner geringen Flüch[illegible] ohne Benutzung eines [illegible] nen Anlagen verar[illegible]

Dangerous, not acceptable!

H. Eckert; (1984); DE 3440

… Furthermore, the use [illegible] surprisingly additional advantages. For example, [illegible] can be distilled without decomposition at 203 – 207 °C [illegible] is almost no reaction with conc. sulfuric acid or cold sodium hy[illegible]oxide solution. **DUE TO THE LOW VOLATILITY TRIPHOSGENE CAN BE USED WITHOUT A FUME HOOD AND, WERE APPLICABLE, IN OPEN PLANT EQUIPMENT. …**

TOXICOLOGY

INHALATION (MAIN ROUTE FOR UPTAKE) RESULTS IN INJURIES IN THE LOWER RESPIRATORY TRACT AND AIRWAYS.

LETHAL CONCENTRATION LC_{50} : 41.5 mg/m³ or 3.4 ppm (LC_{50} of phosgene: 7.2 mg/m³ or 1.8 ppm)

THE VAPOR SATURATION CONC. OF BTC AT 20 °C IS CA. 100 TIMES ITS LC_{50} VALUE (!)

BTC EXHIBITS A BIPHASIC MORTALITY PATTERN (typical for irritant gases, acute toxic effect and a second mortality peak after 11–14 days)

OCCUPATIONAL EXPOSURE LEVEL (OEL) NOT ESTABLISHED (lack of studies)

BTC DOES NOT HAVE THE SAME TOXICOLOGICAL PROFILE AS PHOSGENE!
EVEN THOUGH BTC IS A SOLID, RELEVANT TOX CONCENTRATIONS CAN BE REACHED!
A MORE CONSERVATIVE RISK ASSESSMENT IS NECESSARY (LACK OF DATA!)

MONITORING (DETECTORS)

All available detection devices for phosgene indicate also BTC. It is common practice to use phosgene detectors for BTC handling. However, the DETECTORS ARE CALIBRATED FOR PHOSGENE, ONLY!

Normally, BTC AND PHOSGENE ARE BOTH PRESENT (reaction, off-gas) → inconclusive detector-reading!

BTC-vapor has (as phosgene) a high density and therefore less potential to spread. As result, a delay of detection might occur (potential for sudden & significant concentration changes!)

Regular checks of locations with potential of leakage by phosgene indicator paper is recommended (analogous to phosgene)

COMPARED TO PHOSGENE THE SAFETY CONCEPT NEEDS TO BE MORE CONSERVATIVE

MONITORING (BADGES FOR PERSONAL DOSE MONITORING)

MEDICAL & INDICATOR BADGES ARE INDICATING BTC – BUT THEY ARE CALIBRATED FOR PHOSGENE, ONLY

THE BADGES CANNOT DIFFERENTIATE BETWEEN BTC AND PHOSGENE !

BTC and phosgene are normally interconnected → inconclusive badge reading!

Option: Medical badge (treatment options) **+** indicator badge (for early warning).

1/16 LC_{50}	1/8 LC_{50}	1/4 LC_{50}	1/2 LC_{50}	LC_{50}	
55 ppm*min	111 ppm*min	221 ppm*min	443 ppm*min	885 ppm*min	6148 ppm*min

Indicator and Medic badge
BTC, 74 ppm*min, 1/12 LC50)

PHOTOS TAKEN FROM A PRELIMINARY NON-VALIDATED LAB TEST WITH BTC

LC_{50}:rat, 4h exposure

AFTER EXPOSURE: CONSERVATIVE TREATMENT IS REQUIRED (PHOSGENE SCHEME SHOULD REMAIN THE BASIS)

USERS AND LOGISTICS

FINE CHEMICAL INDUSTRY (INCL. AGROCHEMICALS & PHARMACEUTICALS INTERMEDIATES!), ONLY

COMPANIES USING BTC ARE ALREADY DOMINATING THE CHINESE PHOSGENATION MARKET (FINE CHEMICALS)

CORROSION IS AN ISSUE FOR MOST BTC – MANUFACTURING AND CONSUMING PLANTS

EXPERIENCE FROM COMMUNICATIONS:

- **ONLY BASIC AND LIMITED KNOWLEDGE ABOUT CHEMICAL AND TOXICOLOGICAL PROPERTIES OF BTC IS CURRENTLY AVAILABLE.**
- **MORE EXPERIMENTAL STUDIES ON BTC REACTIVITY ARE DESIRED**

BTC-USERS SHOW A REMARKABLE DIVERSITY

BTC TRANSPORTATION

Shipment is ranging from research quantities to full container loads worldwide by sea and by air.
Bags/drums: 25 kg - 600 kg; transport: truck, ship, rail (& airways)

Because of long transit and/or storage times, BTC (and in case of low quality BTC also decomposition products, e.g., diphosgene) COULD LIKELY BE FOUND BETWEEN THE AIR-SPACE OF THE PRIMARY AND SECONDARY PACKAGING MATERIALS.

In this scenario, BTC MIGHT AFFECT THE INTEGRITY OF THE OUTER PACKAGE (e.g., metal drum) and COULD POSE A SERIOUS HEALTH RISK FOR WORKERS OPENING THE SECONDARY PACKAGING MATERIAL while assuming that the primary packaging material will protect them from exposure.

PROPER RISK ASSESSMENT IS REQUIRED

THE USE OF BTC ON PILOT AND INDUSTRIAL SCALE (1)

THE SAFTEY CONCEPT MUST BE AT LEAST AS STRICT AS FOR PHOSGENE.

SAFETY ASPECTS MUST BE INDIVIDUALLY EVALUATED FOR EACH PROCESS.

AN APPROPRIATE EMPLOYEE TRAINING PROGRAM MUST BE IN PLACE.

PROPER PERSONAL PROTECTIVE EQUIPMENT, **PHOSGENE DOSIMETER BADGES (MEDICAL BADGES) REQUIRED**. Breathing protection is mandatory where contact with BTC/phosgene is possible. For minor operations (e.g., opening of flushed equipment) full-face filter masks might be acceptable. For all other operations, the use of self-contained breathing apparatus (SCBA) is highly recommended.

MONITORING FOR PHOSGENE IS MANDATORY FOR ALL PLANT AREAS, INCLUDING THE STORAGE AREA.

BTC SHOULD BE SEGREGATED FROM OTHER MATERIALS. **ONLY THE CURRENTLY REQUIRED BTC QUANTITIES SHOULD BE STORED. THE STORAGE AREA SHOULD BE VENTILATED TO A SCRUBBER (ON DEMAND).**

THE USE OF BTC ON PILOT AND INDUSTRIAL SCALE (2)

PIPES CONTAINING BTC/PHOSGENE SHOULD BE DISTINCTIVELY MARKED.
Proper material selection is required for plant hardware exposed to BTC (caution: BTC slowly penetrates through PTFE; however, this is rather a corrosion than an occupational health issue). A proper preventative maintenance program should be in place.

ACCUMULATION OF BTC AND/OR PHOSGENE SHOULD BE AVOIDED AS MUCH AS POSSIBLE.

THE PREPARATION OF SOLUTIONS OF BTC IS RECOMMENDED ONLY IN CLEAN, ANHYDROUS SOLVENTS. Bases, Lewis acids (e.g., $FeCl_3$ or $AlCl_3$) or porous substances should not be added to a concentrated solution of BTC because doing so might result in uncontrolled decomposition, releasing large amounts of phosgene.

DEPENDING ON THE DEPHOSGENATION CONCEPT, THE WATER/MOISTURE CONTENT OF THE NITROGEN FLOW SHOULD ALSO BE CHECKED. The scrubber system should also be considered a potential source of water (vapor).

PHOSGENE CAN USUALLY BE DETECTED IN THE OFF-GASES AND ALL LAYERS (organic and aqueous!); this possibility should also be considered in cases of reactor cleaning, etc. **THE ORGANIC PHASE CAN CONTAIN SIGNIFICANT AMOUNTS OF PHOSGENE AND BTC.**

TRIPHOSGENE MSDS

EMERGENCY OVERVIEW

Very toxic by inhalation. Contact with water liberates toxic gas. Causes burns.

Potential Health Effects

Eye: May cause eye irritation. Causes eye burns. Lachrymator (substance which increases the flow of tears).

Skin: May cause skin irritation. Causes skin burns.

Ingestion: May cause irritation of the digestive tract. Causes gastrointestinal tract burns.

Inhalation: May cause respiratory tract irritation. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Toxic if inhaled. May cause burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.

Chemical Stability:	Moisture sensitive. Heat sensitive.
Conditions to Avoid:	Incompatible materials, excess heat, exposure to moist air or water.
Incompatibilities with Other Materials	Strong oxidizing agents, acids, strong bases, amines, iron oxide, activated carbon.
Hazardous Decomposition Products	Hydrogen chloride, chlorine, phosgene, carbon monoxide, oxides of phosphorus, carbon dioxide.
Hazardous Polymerization	Has not been reported.

Section 11 - Toxicological Information

RTECS#:	CAS# 32315-10-9: None listed
LD50/LC50:	RTECS: Not available. Other: Oral rat LD50 > 2000 mg/kg Skin rat LD50 > 2000 mg/kg
Carcinogenicity:	Triphosgene - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.
Other:	The toxicological properties have not been fully investigated.

TRIPHOSGENE – SUMMARY

SOMETIMES VERY MISLEADING LITERATURE → WRONG & DANGEROUS !

THE TOXICOLOGICAL PROFILE OF BTC IS DIFFERENT FROM THAT OF PHOSGENE

THERE ARE NEITHER DETECTION DEVICES NOR BADGES DEDICATED TO BTC

USE OF BTC IS ALREADY A STANDARD APPROACH IN FINE CHEMICALS INDUSTRY IN CHINA 

AND A SLOWLY INCREASING ONE IN INDIA 

PRINCIPLE SAFETY AWARENESS IS NORMALLY EXISTING AMONGST USERS

TECHNICAL STANDARDS (CHINA & INDIA) ARE NORMALLY BETWEEN VERY PROBLEMATIC AND ACCEPTABLE

TRIPHOSGENE (BTC) HAS ADVANTAGEOUS FEATURES – ESPECIALLY FOR SMALL – AND MEDIUM – SCALE PHOSGENATIONS

SAFE HANDLING IS POSSIBLE – BUT BTC IS NOT A "SAFE PHOSGENE"!

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